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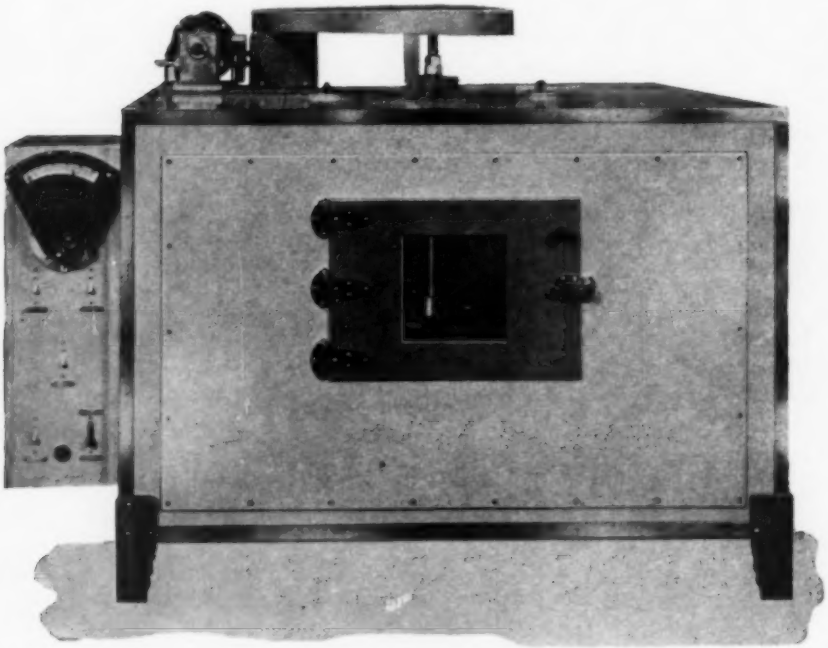

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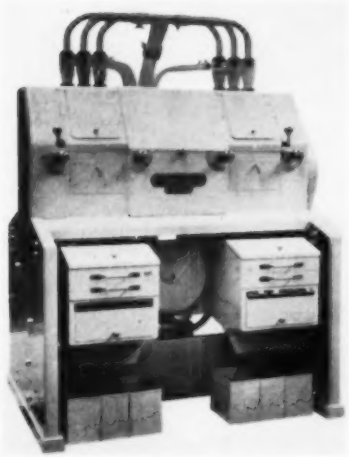

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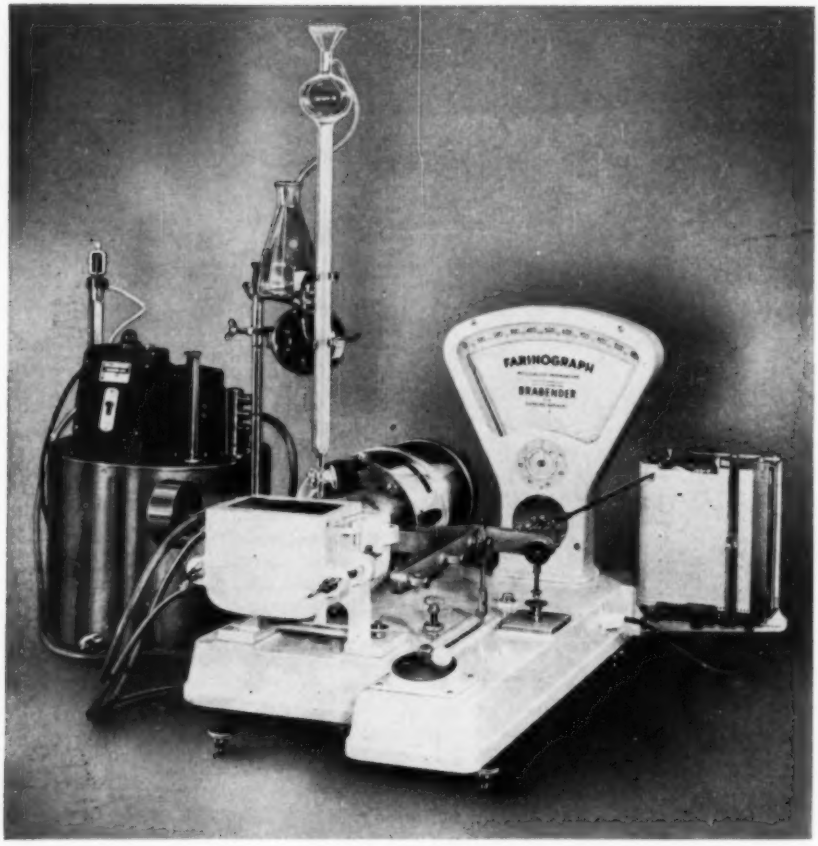

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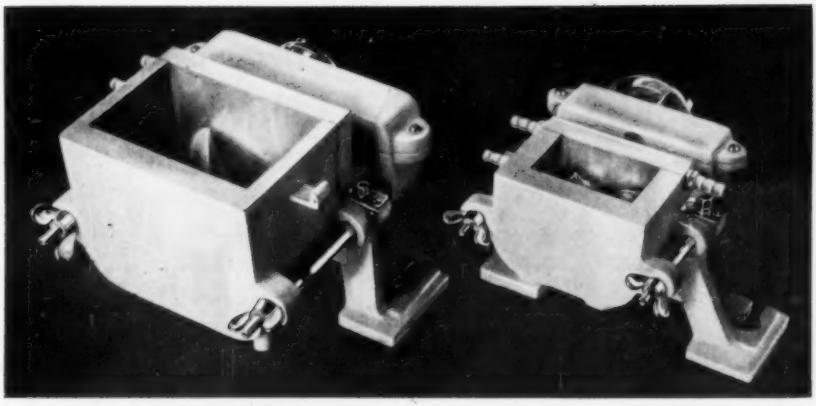

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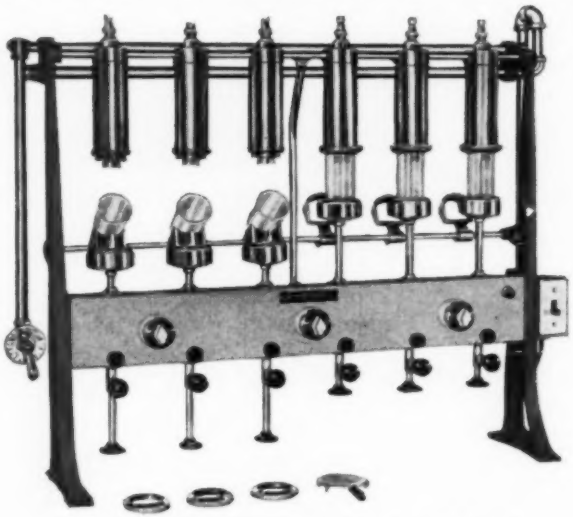

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CEREAL CHEMISTRY

VOL. 33 MARCH, 1956 NO. 2

A STUDY OF THE DESORPTION ISOTHERMS OF WHEAT AT 25°C. AND 50°C.[1]

H. A. BECKER AND H. R. SALLANS

ABSTRACT

Desorption isotherms for wheat have been determined at temperatures of 25° and 50°C. Differential net heats of desorption calculated from the isotherms indicate that the initial heat is in the order of 7–10 k. cal. per mole of water desorbed and that the differential heat becomes negligible in comparison with the normal heat of vaporization of water at moistures much greater than 30%, dry basis. Heats of dehydration have been computed and indicate that the total heat of dehydration is approximately 43–47 cal. per g. of wheat, dry-matter basis. Average values of the net heat of desorption to be used in drying calculations have been computed. It is also shown that there is an evolution of heat when a drying wheat kernel is allowed to shift from nonuniform to uniform moisture distribution at constant average moisture content, and the magnitude of this effect is evaluated.

Water sorption equilibria in wheat and wheat products have been studied by numerous investigators (7, 8), and Babbitt (2) was among the first to distinguish between adsorption and desorption equilibria in wheat. However, no rigorous investigation of the effect of temperature on the equilibrium has yet been made. In the present study desorption isotherms were determined at two different temperatures and the heats of desorption calculated. The results have some interesting implications relevant to grain drying problems.

Materials and Methods

Wheat. The wheat was from the same lot used by the authors in drying studies (4). It was tempered to an initial moisture content of 32%, dry basis, and then stored for 10 days at 10°C. to induce moisture equilibration in the kernels. An initial moisture of 32% was chosen because preliminary tests indicated that this is the transition region from "bound" to "free" water.[2] When the wheat is saturated with "bound" water any errors due to hysteresis should be minimized. Initial moistures higher than 32% were avoided because of the danger of mold, irreversibilities in swelling, etc.

[1] Manuscript received November 21, 1955. Contribution from the Prairie Regional Laboratory, National Research Council, Saskatoon, Saskatchewan. Issued as N.R.C. No. 3879 and No. 332 of the Associate Committee on Grain Research (Canada).

[2] It is assumed that "free" water can be removed by the normal heat of vaporization, whereas "bound" water requires heat in excess of this value.

The wheat was coarsely ground in a Hobart mill to hasten the approach of desorption equilibrium and so reduce the danger of mold growth in samples exposed to high humidities. The investigation of Babbitt (2) indicates that coarse subdivision has no effect on *desorption* equilibria.

Wide-mouth jars 10 cm. high and 5 cm. in diameter were filled one-third full with sulfuric acid solutions to give atmospheres of constant humidity. Wheat samples (1.5 g.) were placed in 2-cm. cubical baskets formed from fine stainless-steel screen and suspended in the jars. The jars were then placed in a constant-temperature cabinet.

A period of 10 days was found sufficient for moisture vapor equilibrium and was used for all experiments. The jars were shaken each day for the first 3 days to thoroughly mix the sulfuric acid solutions. After equilibration of the samples, moisture contents were determined by the 130°C., 1-hour oven method (1). At moisture levels of 15% and higher the maximum deviation in replicates by this method was ±0.1%; at 12% and lower the deviation was ±0.03%. All moisture contents are expressed on a dry-matter basis.

Final concentrations of the sulfuric acid solutions were determined by titration with standard alkali and by determination of specific gravity. The two methods were in good agreement. The water vapor pressures were computed from tables given by Perry (9).

Results

The desorption equilibria between vapor pressure of water and moisture content of wheat were studied at 25° and 50°C. The data are given in Table I, where m is the moisture content, g/g dry-matter basis,[3] and f is the relative vapor pressure of water. The determinations were done at the time that lot "B" was being studied in drying experiments (4), and the wheat samples were therefore in comparable condition.

The determination at zero relative vapor pressure was made by desiccating a sample of ground wheat over magnesium perchlorate for 3 weeks. At the end of this time the wheat still contained 1.25% moisture. Babbitt (2), studying a system under high vacuum, found that whole-wheat kernels had residual moisture contents of 3.0% at 25°C. and 0.8% at 34°C. When flour was used under similar conditions the moisture was completely removed. Hence it appears that the value for moisture content at zero relative humidity reflects a lack of complete equilibrium rather than any abnormal conditions of adsorption.

Desorption Isotherms. The desorption isotherm of wheat is sigmoid,

[3] All moisture contents are expressed on dry-matter basis unless specified otherwise.

TABLE I

MOISTURE-VAPOR PRESSURE DESORPTION EQUILIBRIUM DATA FOR WHEAT

25°C.		50°C.	
f RELATIVE VAPOR PRESSURE, WATER	m MOISTURE, G/G DRY MATTER	f RELATIVE VAPOR PRESSURE, WATER	m MOISTURE, G/G DRY MATTER
0.000	0.0125[a]	0.127	0.0471
0.045	0.0421	0.206	0.0609
0.090	0.0602	0.286	0.0698
0.111	0.0646	0.396	0.0907
0.203	0.0830	0.596	0.1222
0.328	0.1068	0.669	0.1411
0.520	0.1395	0.757	0.1610
0.683	0.1660	0.823	0.1878
0.747	0.1852	0.884	0.2238
0.863	0.2351	...	...
0.930	0.2810	...	...

[a] Obtained by desiccating a wheat sample for 3 weeks above magnesium perchlorate.

of type III according to the classification of Brunauer, Emmett, and Teller (5). The features of the isotherm have been described recently (7) and consist of three portions: an initial curved section which is concave downward, a linear region, and finally a curved section which is concave upward. The purpose of the present study is to define as closely as possible the relation between moisture content and relative vapor pressure in each region. This was done as follows:

(i) Many biological materials follow the Brunauer, Emmett, Teller (B.E.T.) equation (5) in the initial curved portions of their water sorption isotherms, and it has been implied by Hlynka and Robinson (7) that the equation also describes the behavior of wheat in this region. In terms of moisture content, dry matter basis, the B.E.T. equation is $m/m_m = cf/(1-f)\,(1+(c-1)\,f)$, where m_m is the moisture content required to form a unimolecular layer and c is a constant approximately equal to exp. H_m/RT; where H_m is the average net heat of desorption of the unimolecular layer, R is the gas constant, and T is the absolute temperature. According to the equation a plot of $f/m\,(1-f)$ against f should be a straight line with a slope of $(c-1)/cm_m$ and an intercept of $1/cm_m$. Figure 1 shows B.E.T. plots of the desorption equilibrium data for wheat at the two temperatures studied. It appears that at 25°C. the relation plotted is truly linear up to a relative vapor pressure of about 0.35; this is the usual upper limit of applicability of the simple B.E.T. equation. There were not sufficient data to prove the linearity of the plot at 50°C., and the curve

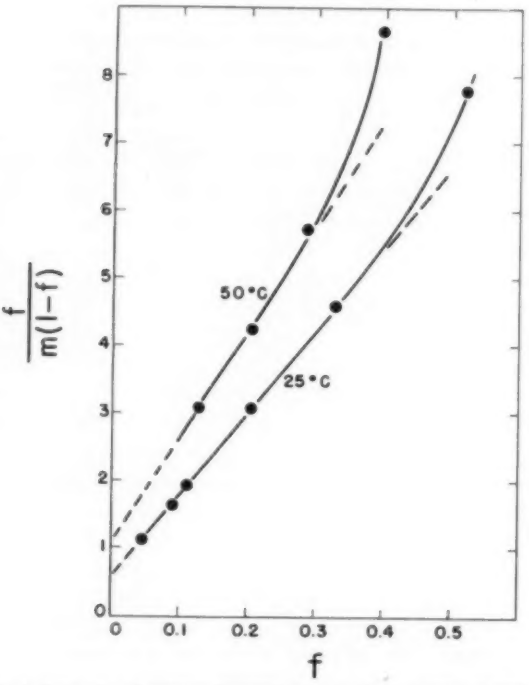

Fig. 1. B.E.T. plot of moisture desorption data on wheat at 25° and 50°C.

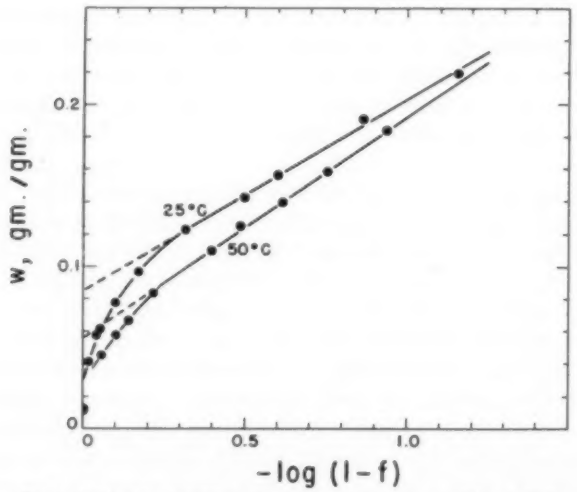

Fig. 2. Smith plot of moisture desorption data on wheat at 25° and 50°C.

shown in Fig. 1 for this temperature has been drawn on the assumption that the equation is followed up to a relative vapor pressure of about 0.3.

(ii) It has been shown by Smith (11) that the final curved portions of the water sorption isotherms of biological materials such as starch, zein, and cellulose are closely described by the equation

$$w = w_b - w' \ln (1 - f),$$

where w is the moisture content, g/g, *wet basis*, w_b is the intercept in a plot of w *vs.* $\ln (1 - f)$, and w′ is the slope. Figure 2 shows a Smith plot of the desorption equilibrium data for wheat. In the range of relative vapor pressures 0.5 to 0.95 the relation plotted is essentially linear at both temperatures studied, indicating that the Smith equation describes the desorption isotherm in this region.

(iii) The intermediate region of the desorption isotherm is apparently linear. The following method, illustrated in Fig. 3, for defining the curve in this region, was found to result in an excellent fit of the experimental data and gave a smooth unbroken transition into the curved regions described by the B.E.T. and Smith equations: a straight line was drawn tangential to the Smith curve to intersect the B.E.T. curve at its point of inflection.

Table II shows a summary of the isotherm equations and gives

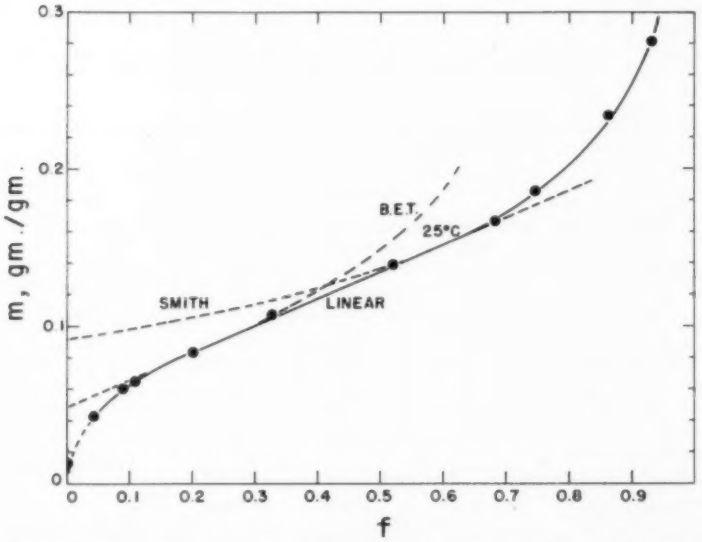

Fig. 3. Desorption isotherm of wheat at 25°C., showing the method of obtaining the linear region from the curves of the B.E.T. and Smith equations.

the values of the constants at 25° and 50°C. as determined from the desorption equilibrium data for wheat. The table also indicates the approximate regions of applicability of the equations. Using the equations, complete desorption isotherms giving the best fit of the experimental data were drawn and are shown in Fig. 4.

Differential Net Heat of Desorption. The differential net heat of desorption – the heat required to remove one mole of water from an

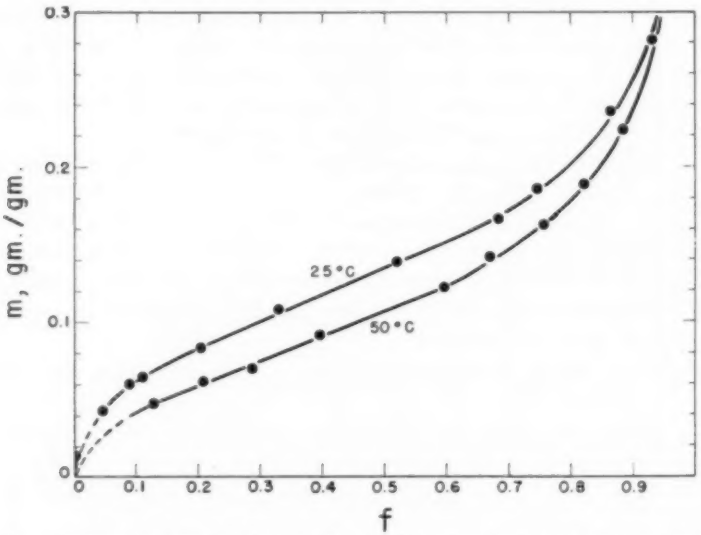

Fig. 4. Desorption isotherms of wheat at 25° and 50°C.; curves drawn according to the isotherm equations found applicable in each region.

TABLE II

DESORPTION ISOTHERM EQUATIONS AND THE VALUES OF THEIR CONSTANTS FOR WHEAT AT 25° AND 50°C.

Equation	Values of Constants			Approximate Region of Applicability	
	Const.	25°C.	50°C.	25°C.	50°C.
1. B.E.T. equation:	m_m	0.0780	0.0602	$0.04 < f < 0.30$	
$\frac{m}{m_m} = \frac{cf}{(1-f)(1+(c-1)f)}$	c	22.9	15.1		
2. Linear equation:	m_o	0.0483	0.0278	$0.12 < f < 0.65$	
$m - m_o = af$	a	0.1715	0.1564		
3. Smith equation:	w_b	0.0845	0.0550	$0.50 < f < 0.95$	
$\frac{w - w_b}{w'} = \ln \frac{1}{1-f}$	w'	0.0514	0.0595		

infinitely large weight of adsorbent—can be obtained from the well-known thermodynamic equation

$$\overline{H} = (H/n) = (RT_1T_2/T_2 - T_1) \ln (f_2/f_1),$$

where f_2 is the relative vapor pressure at the higher temperature T_2 which corresponds to the same moisture content as the relative vapor pressure f_1, at the lower temperature T_1. In computing the differential heat from this equation the values of f_1 and f_2 at a given moisture level can be obtained from the isotherm equations for the two temperatures. The differential heat is expressed in cal. per mole of water removed.

Figure 5 shows the differential net heat of desorption at 25°C. as a function of moisture content. Extrapolation beyond the data to zero moisture content using the B.E.T. equation gave a result of doubtful significance; i.e., at moisture levels below 5% the differential heat decreased with decreasing moisture content. Since this type of behavior is unlikely it is evident that the B.E.T. equation is not applicable at relative vapor pressures below about 0.04. Extrapolation beyond the data at high moistures by the Smith equation produced a smooth continuation of the differential heat curve. The Smith equation predicted that the differential heat should be zero at a moisture content of about 37%; this corresponds to the point of intersection of the isotherms. The Smith equation is not reliable beyond a relative vapor

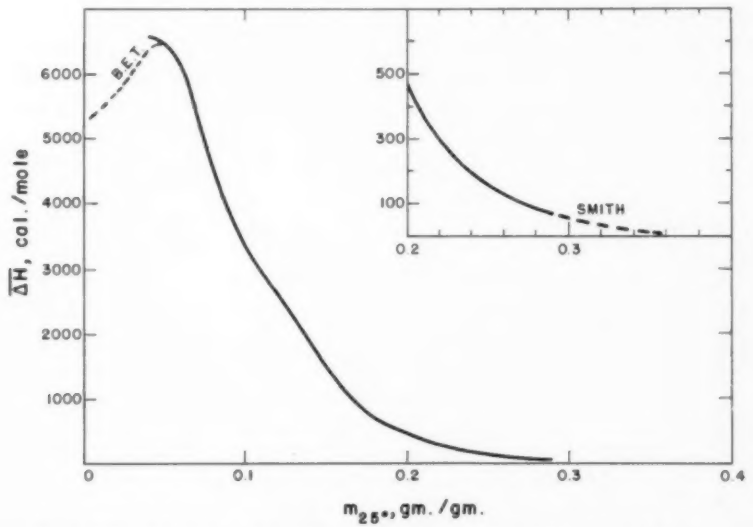

Fig. 5. Differential net heat of desorption of water from wheat at 25°C., computed from the isotherms at 25° and 50°C. and shown as a function of moisture content, dry basis.

pressure of 0.95, corresponding to about 30% moisture at 50°C.; but it appears that the differential heat *approaches* zero in the region of 37% moisture.

The differential heat curve shows a regular decay of the binding energy with increasing moisture content, emphasizing the indefiniteness of the concept of "bound" water when applied to substances such as wheat. It would appear from Fig. 5 that the initial heat is quite high, probably of the order of 7 k. cal. per mole and certainly not greater than 10 k. cal. per mole. The high initial heat probably results from chemisorption on polar groups, possibly by hydrogen bond formation. The values of the differential net heat of desorption for wheat are closely comparable with the values of the differential net heat of sorption computed by Dole and McLaren (6) for the protein collagen, except that with collagen the differential heat remains appreciable up to much higher moisture levels.

Heat of Dehydration. The heat of dehydration is defined as the additional heat above the normal heat of vaporization required to reduce the moisture content from saturation to a designated level, and is expressed in cal. per g. of wheat, dry basis. Figure 5 indicates that only a moisture content below a level of 37% will make a significant contribution to this value. The heat of dehydration can be obtained by integrating the area under the differential heat curve from any designated moisture level up to a point where the differential heat approaches zero (37%).

Figure 6 shows the heat of dehydration of wheat as a function of moisture content. Since the computed heat at 30% moisture is only 0.1 cal. per g., any error occasioned by extrapolation beyond the data

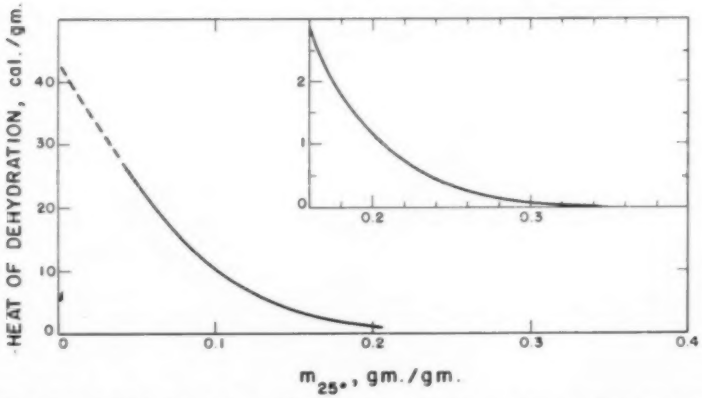

Fig. 6. Heat of dehydration of wheat at 25°C., shown as a function of moisture content, dry basis.

to high moistures becomes negligible. In extrapolating to zero moisture content it was assumed that the differential heat at zero moisture is 7 k. cal. per mole, giving a total heat of dehydration of 43 cal. per g. An initial differential heat of 10 k. cal. per mole would give a total heat of dehydration of about 47 cal. per g., which is only 9% higher. Thus the present results indicate that the total heat of dehydration is in the order of 43 cal. per g. and should not be higher than 47 cal. per g.

Equilibrium Net Heats of Desorption in the Drying of Wheat. From the heats of dehydration or from the differential heat curve, average values of the net heat of desorption can be computed. Table III shows a series of values so obtained. The net heat is expressed in B.T.U. per lb. of water removed, and is an average value to be added to the normal heat of vaporization of water when drying wheat in the ranges of moisture reduction indicated. It should be emphasized that these are *equilibrium* heats of desorption and are valid only when the initial and final moisture distributions in the kernel are uniform.

TABLE III

AVERAGE EQUILIBRIUM NET HEATS OF DESORPTION IN THE DRYING OF WHEAT

INITIAL MOISTURE % WET BASIS	AVERAGE NET HEAT OF DESORPTION, B. T. U. PER POUND	
	Final Moisture = 13%, Wet Basis	Final Moisture = 14%, Wet Basis
15	110	94
16	96	81
17	83	70
18	73	61
19	64	53
20	57	47
21	52	42
22	46	38

Thermal Effects of Nonuniform Moisture Distribution in Drying Wheat Kernels. In a drying wheat kernel there is a moisture gradient from the interior of the kernel to the surface. From the nature of the differential heat curve there must also be a difference in desorption energy levels between uniform and nonuniform distributions. To estimate the magnitude of this difference it is necessary to determine (a) the moisture gradient in the drying kernel, (b) the corresponding distribution in heats of dehydration, and (c) the average heats of dehydration for uniform and nonuniform moisture distributions.

The moisture gradient in a drying wheat kernel assumed to be

spherical can be calculated from the following equation given by Barrer (3):

$$\frac{m - m_s}{m_o - m_s} = \sum_{n=1}^{\infty} \frac{1}{n} (-1)^{n+1} \sin (n\, r/r_o) \exp (-n^2\pi^2 D_c t/r_o^2),$$

where r_o is the radius of the kernel,

m_o is the initial uniform moisture content (all moisture contents are expressed on dry-matter basis)

m is the moisture content at a distance r from the center at a time t,

m_s is the moisture content at the surface of the kernel, at $r = r_o$, and

D_c is the diffusion coefficient.

Furthermore, the average moisture content at time t is given by the equation:

$$\frac{\bar{m} - m_s}{m_o - m_s} = \frac{6}{\pi^2} \sum_{n=1}^{\infty} \frac{1}{n^2} \exp (-n^2\pi^2 D_c t/r_o^2)$$

This equation has been used to calculate diffusion coefficients from drying data. The authors (4) used the notations

$$\overline{M} = (\bar{m} - m_s)/(m_o - m_s) \quad \text{and} \quad D't = \pi^2 D_c t/r_o^2$$

and have given general solutions of the equation in a graph showing $\overline{M}$ as a function of $D't$. In the equation for the moisture gradient let the notation $M = (m - m_s)/(m_o - m_s)$ be used. Inspection of the equations shows that the moisture gradient and $\overline{M}$ are unique functions of $D't$, and for each value of $\overline{M}$ there exists a unique solution of the moisture gradient which can be expressed by showing $\overline{M}$ as a function of r/r_o.

The equations were solved for a number of values of $D't$ and Fig. 7 shows the resulting moisture gradients with the corresponding values of $\overline{M}$. For calculation of the thermal effect in shifting from non-uniform to uniform moisture distributions the solution for $\overline{M} = 0.500$ is most suitable. The following moisture values were therefore selected:

(i) $\bar{m} = 0.163$ g/g, or 14% wet basis, the usual level to which wheat is dried;

(ii) $m_s = 0.103$ g/g, the value found in drying thin layers of wheat in a current of hot air (4);

(iii) $m_o = 0.223$ g/g, or 18.2% wet basis, a reasonable value for damp wheat: m_o is by the definition of $\overline{M}$, an automatic result of the specification of values for $\bar{m}$, m_s, and $\overline{M}$.

Using these values the absolute moisture distribution in the kernel was calculated and the corresponding heats of dehydration evaluated.

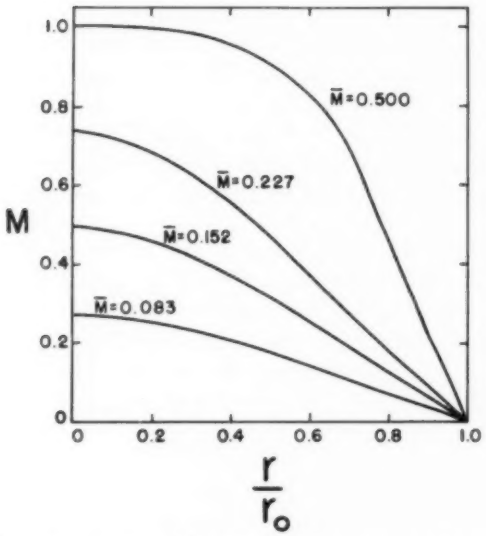

Fig. 7. Moisture gradients in drying wheat kernels calculated from the equations for diffusion out of spheres.

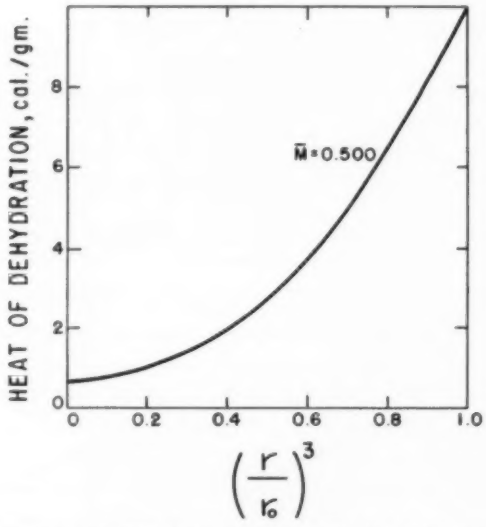

Fig. 8. Nonuniform distribution of the heat of dehydration in a drying wheat kernel.

Figure 8 was then prepared, showing the heat of dehydration as a function of $(r/r_o)^3$, where $(r/r_o)^3$ is proportional to volume and hence,

in the present case, gives the weight average distribution of the heat of dehydration. In this graph the area under the curve gives the average value of the heat of dehydration for the nonuniform distribution. The value found was 3.65 cal. per g. From Fig. 6 the heat corresponding to a uniform moisture distribution of 0.163 g/g (or 14%, wet basis), is 2.65 cal. per g., and hence the shift to an equilibrium distribution is accompanied by an evolution of heat equal to $3.65 - 2.65 = 1.00$ cal. per g.

The difference between heats of dehydration for uniform and nonuniform moisture distributions leads to some interesting conclusions. First, since the heat is greater for a nonuniform distribution there is an increase in the average heat of desorption in drying. For drying from 18.2 to 14%, wet basis, the moisture removed is 0.060 g/g, dry basis, and the increase is 31 B.T.U. per lb. of moisture removed. This amount must be added to the equilibrium heat in Table III, giving for the total heat $61 + 31 = 92$ B.T.U. per lb. Secondly, when this wheat is put in a bin or thermos flask, and is allowed to come to an equilibrium moisture distribution, there is an evolution of heat equal to 1.00 cal. per g. dry basis or 0.87 cal. per g. wet basis. The specific heat of wheat at 14% moisture content, wet basis, is 0.414 cal. per g. °C. (10), and if the wheat is stored under conditions of no heat or moisture loss, as in a thermos flask, the heat evolved will raise the temperature of the grain by $(0.87)(1.8)(0.414) = 3.8°F$. This effect is of practical importance in measuring grain temperatures; the temperature should be measured immediately after the grain is removed from the dryer and as rapidly as possible (4). It also serves to emphasize the necessity and importance of a cooling section in any practical grain dryer.

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THE EFFECT OF SUCROSE ON THE PROPERTIES OF SOME STARCHES AND FLOURS[1]

E. ELIZABETH HESTER,[2] ALICE M. BRIANT,[3] AND CATHERINE J. PERSONIUS[3]

ABSTRACT

The effect of four concentrations of sucrose on some paste and gel properties of a corn starch, two wheat starches, two wheat flours, and two nongelling waxy-corn starches has been investigated.

In the presence of sucrose, the hydration of starch granules was inhibited (decreased) as evidenced by the following: 1) the temperature of the initial rise in paste viscosity was raised for most of the starches, indicating delayed swelling of the granules; 2) the maximum viscosity of the starch pastes was lower or not reached at 95°C., indicating less swelling of the granules; 3) the disintegration of the granules was less, and either the granule size or the extent of implosion and folding of the granules was decreased; 4) the amount of soluble material diffusing from the starch granules was less; and 5) the starch gels decreased in rigidity and, when high sucrose concentrations were used, gels were not formed.

The pastes of the wheat flours heated in the presence of sucrose were markedly higher in maximum and terminal viscosity and the gels tended to be firmer. Comparable pastes of the wheat starches showed no maximum viscosity and little or no change in terminal viscosity in the presence of sucrose, and the gels were weakened. Apparently the effect of sucrose on constituents of the flour, other than starch, influenced these paste and gel properties.

The characteristics of starch sols and gels may be altered markedly by the presence of materials other than starch. Numerous reports appear in the literature on the effect of pH, fat acids, emulsifying agents, and various inorganic salts on the behavior of starch. Although sugar is a common ingredient of starch-thickened food mixtures, few studies of the effect of sugar on starch are reported.

The work described herein concerns the effect of sucrose on some paste and gel properties of a corn starch, two wheat starches, two wheat flours, and two nongelling waxy-corn starches. The physical

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properties studied were the pasting characteristics and the viscosity of the hot pastes; the rigidity and water-holding capacity of the gels; and the number and size of swollen granules for a given weight of dry starch. Chemical determinations of the soluble starch and its approximate composition were made on the corn and wheat starch pastes.

Materials and Methods

Commercially prepared corn starch[4], waxy-corn starch[4], modified waxy-corn starch[4], a hard and a soft wheat flour, and starches prepared in the laboratory from these flours were studied. The modified waxy-corn starch was a cross-linked starch prepared by reacting unruptured waxy-corn starch granules with a polyfunctional reagent, phosphorus oxychloride.[5]

The wheat flours were an unbleached New York State soft-wheat pastry flour and an unbleached long patent hard red spring wheat flour. Starches were prepared from these flours by water extraction from a stiff dough, followed by air drying.

The following concentrations of starch and flour in water gave gels of comparable rigidity at 30°C.: 6.5 g. corn starch, 7.3 g. hard-wheat starch, 7.5 g. soft-wheat starch, 11.3 g. soft-wheat flour, and 13.8 g. hard-wheat flour per 100 g. water. The concentrations of the waxy starches used gave pastes comparable in terminal viscosity to the corn starch; they were 4.9 g. modified waxy- and 5.3 g. waxy-corn starch per 100 g. water.

The sucrose was a commercial grade of fine granulated cane sugar. The amounts used were 15.9, 23.7, 31.6, and 39.5 g. per 100 g. water, and represent amounts commonly used in puddings and cream pie fillings.

Preparation of and Measurements on the Pastes and Gels. The starch or flour was suspended in distilled water at 25°C. and transferred quantitatively to the bowl of a Brabender Amylograph, the cooling coil of which had been removed. When sucrose was used, the dry sugar was combined with the dry starch or flour before the water was added. Viscosity-temperature curves were recorded from 25°C. to 95°C. Even though maximum paste viscosity might not have been attained, heating was terminated at 95°C., thus providing constant heating conditions for all pastes.

Rigidity or sag of the corn starch, wheat starch, and flour gels was measured by the Exchange Ridgelimeter with the method described by Cox and Higby (2) for pectin gels. For this test a portion of hot

[4] American Maize Products Co., Roby, Indiana.
[5] Felton, George E., and Schopmeyer, Herman H., U.S. Patent 2,328,537 (1943).

paste was poured into jelly glasses with sideboards, covered, and stored at 30°C. overnight.

The water-holding capacity of the gels was determined using 50-g. samples of the gelatinized pastes which had been aged for 2 days at about 5°C. The aged gels were placed on a 30-mesh sieve, which was fitted in a short-stem funnel, and covered with a watch-glass. The liquid collected in a 2-hour period is reported as a measure of the water-holding capacity of the gels. To minimize evaporation, aluminum foil was placed around the funnel stem and over the collecting beaker.

Microscopic Measurements. The effect of heat and of added sucrose on the appearance of the starch granules was observed by means of a phase microscope. A hemocytometer and micrometer with calibrated eyepiece were used in counting the number of granules of different sizes. A 10-g. aliquot of the cooked paste was diluted to 1 liter and one drop of this suspension was used to fill the chamber of the hemocytometer. The number of granules in each of six classes, mean diameters ranging from 7 to 67 μ, was counted in four 1-mm. squares. The number of granules per g. of dry starch of each of the sizes and the volume of granules per g. of starch were calculated. The swollen granules were assumed to be spherical.

Estimation of Soluble Starch and Its Composition. Slurries of corn starch and of the two wheat starches were heated to 95°C. in test tubes in a boiling-water bath. The same concentrations of starch, sugar, and water were used as in the pastes prepared in the amylograph. Six volumes of methanol were used to precipitate the soluble material in the supernatant of the washed and centrifuged hot pastes. The mixture was refrigerated for 48 hours and the precipitate collected in sintered Jena crucibles, washed with methanol, and dried for 24 hours over calcium chloride in a vacuum dessicator.

A modified colorimetric iodine-titration method was used to estimate the relative amount of the linear fraction in the soluble starch. The method of preparing the precipitate for the iodine titration was essentially that of Kerr (4). The iodine uptake was measured by the percent light transmitted, using an Evelyn Colorimeter with a 565 mμ filter. Density was plotted against the milliliters of iodine solution added. The uptake of iodine in complex formation was estimated by extrapolation of the two straight-line portions of the curve. The estimated percent of the linear fraction of the soluble starch was calculated on the basis of the iodine affinity. The iodine affinity of pure corn amylose was assumed to be 18.7% (1) and of the wheat amylose, 19.9% (5). It is recognized that calculations based on the iodine

affinity of amylose from one source of a defatted corn or a defatted wheat starch are empirical.

Further details of the methods are given by Hester (3).

Results and Discussion

Gelatinization Characteristics. The marked effect of varying concentrations of sucrose on the gelatinization characteristics of the starch and flour pastes is shown in the amylograms in Figs. 1 and 2.

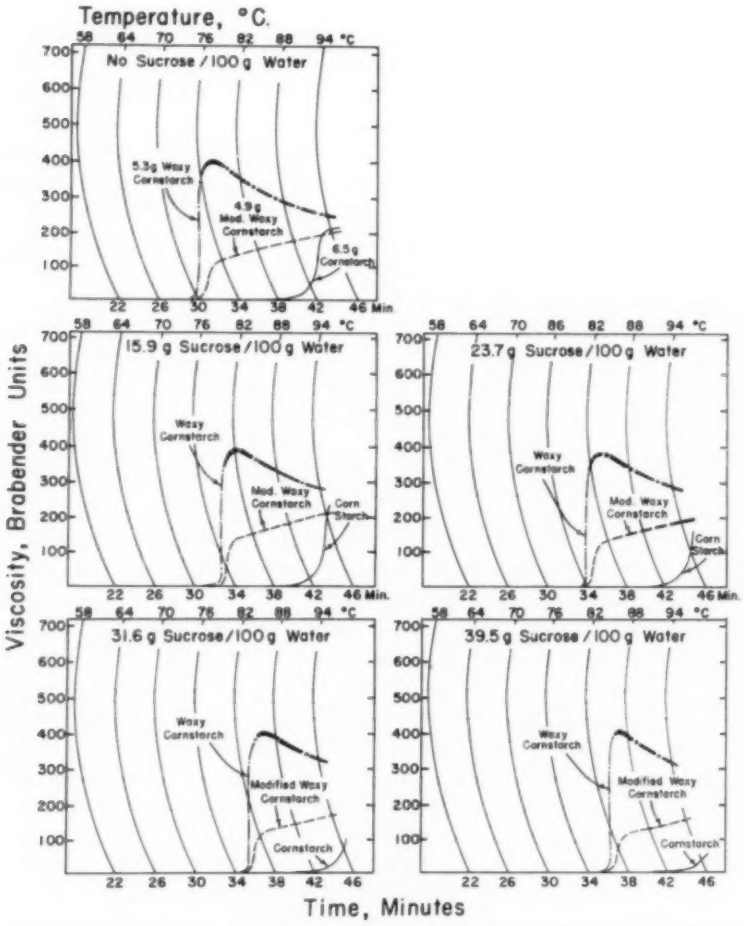
Temperature, °C.
58 64 70 76 82 88 94 °C
No Sucrose / 100 g Water
5.3g Waxy Cornstarch
4.9g Mod. Waxy Cornstarch
6.5g Cornstarch
22 26 30 34 38 42 46 Min
15.9 g Sucrose / 100 g Water
Waxy Cornstarch
Mod. Waxy Cornstarch
Corn Starch
23.7 g Sucrose / 100 g Water
31.6 g Sucrose / 100 g Water
Modified Waxy Cornstarch
Cornstarch
39.5 g Sucrose / 100 g Water
Viscosity, Brabender Units
Time, Minutes

Fig. 1. Amylograms showing the effect of increasing sucrose concentrations on the gelatinization characteristics of corn and waxy-corn starches. The weights of starch are for 100 g. water and were used with each sucrose concentration.

The initial viscosity rise of the waxy-corn starches, of the corn starch, and of the flours occurred at progressively higher temperatures as the concentration of sucrose was increased. The pastes of the wheat starches exhibited little or no change in this respect.

The sharp rise in viscosity of the waxy-corn starch pastes persisted with each increase in sucrose concentration. The temperature at which the maximum paste viscosity occurred increased progressively with in-

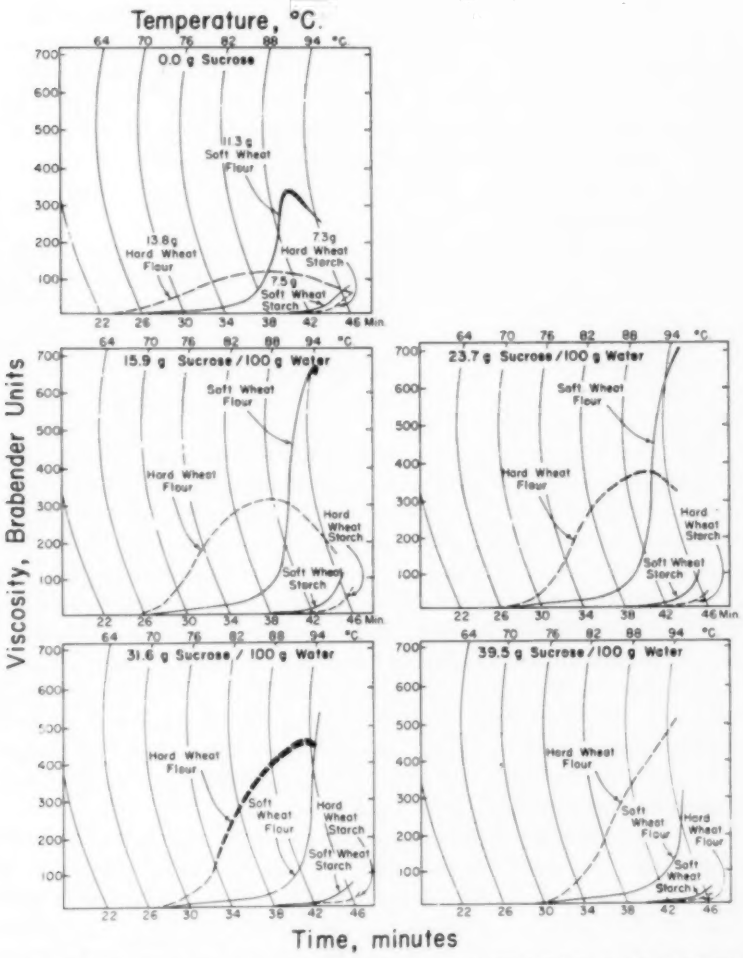
Temperature, °C.
64 70 76 82 88 94 °C
0.0 g Sucrose
11.3 g Soft Wheat Flour
13.8 g Hard Wheat Flour
7.3 g Hard Wheat Starch
7.5 g Soft Wheat Starch
22 26 30 34 38 42 46 Min.
15.9 g Sucrose / 100 g Water
23.7 g Sucrose / 100 g Water
31.6 g Sucrose / 100 g Water
39.5 g Sucrose / 100 g Water
Soft Wheat Flour
Hard Wheat Flour
Hard Wheat Starch
Soft Wheat Starch
Viscosity, Brabender Units
Time, minutes

Fig. 2. Amylograms showing the effect of increasing sucrose concentrations on the gelatinization characteristics of wheat flours and their starches. The weights of starch and flour are for 100 g. water and were used with each sucrose concentration.

creasing sucrose concentration, but the magnitude of the maximum viscosity (about 400 B.U.) was not affected. Less swelling and less disintegration of the granules was observed with increased concentrations of sucrose. With high concentrations of sucrose the decreased granule disintegration apparently obscured the effect of decreased swelling on paste viscosity.

The characteristic shape of the viscosity-temperature curves of the modified waxy-corn starch, and of the corn and wheat starches, was not much altered by the addition of sucrose. The maximum viscosity of most of the pastes had not been reached when the heating was terminated. A progressively lower terminal viscosity resulted with increases of sucrose beyond the lowest concentration. With the low sucrose concentration, possibly the swelling of the granules was not greatly inhibited, or the sucrose itself may have contributed sufficient viscosity to the mixture to mask any effect of delayed hydration of the starch granules.

In general, the higher temperature for the initial viscosity rise and the lower viscosity at 95°C. with increasing sucrose concentrations for all the starches suggest a slower initiation of hydration and a lesser final hydration of the starch granules. Perhaps continued heating at 95°C. would have allowed for greater hydration and so for a greater maximum viscosity than was obtained under the conditions used in this study.

The viscosity-temperature curves of the two flours, particularly of the hard wheat, exhibited the most marked changes in shape. The rise in viscosity of the hard-wheat flour pastes was sharper, the maximum viscosity was higher, and the temperature of the maximum was increased with each addition of sucrose. The maximum viscosity of the soft-wheat flour paste was sharply increased by the addition of only 15.9 g. of sucrose and the temperature at the maximum was higher. The next higher concentration resulted in a further but slight increase in the maximum paste viscosity, but further increases of sucrose caused a decrease. Reduced breakdown in viscosity, or none, was observed in the flour pastes containing sucrose. In the sucrose solution of highest concentration the paste viscosity had not reached maximum when heating was terminated.

In view of the gelatinization behavior of the starches washed from these flours it is doubtful that the effect of sucrose on the starch in the flour could have been a factor contributing to the increased paste viscosity. The protein content of the hard spring wheat flour is greater than that of the soft-wheat flour, and the protein content of the flours offers the most obvious explanation for the differences between the

flours and their starches. The possible role of other constituents of flour should not be overlooked.

Gel Rigidity. The change in rigidity of the starch and flour gels with increasing amounts of sucrose is shown in Table I. The differences attributable to the thickening agents, to the sucrose concentrations, and to their interaction were significant at the 1% level.

TABLE I

RIGIDITY OF STARCH AND FLOUR GELS WITH INCREASING SUCROSE CONCENTRATIONS
(Averages of three replicates)

THICKENING AGENT AND CONCENTRATION (Grams per 100 g. Water)		GRAMS SUCROSE PER 100 G. WATER				
		0.0	15.9	23.7	31.6	39.5
		Sag				
		%	%	%	%	%
Corn starch	6.5	21.6	23.7	coll.[a]	none[b]	none[b]
Hard-wheat starch	7.3	21.5	23.8	coll.[a]	none[b]	none[b]
Soft-wheat starch	7.5	21.2	20.4	47.9[c]	none[b]	none[b]
Soft-wheat flour	11.3	20.9	17.5	17.5	coll.[a]	none[b]
Hard-wheat flour	13.8	21.7	14.6	13.3	11.2	10.9

[a] Gel collapsed.
[b] No gel.
[c] One gel collapsed and was arbitrarily assigned a sag of 100%.

The gels of the corn starch and of the hard-wheat starch decreased slightly in rigidity when prepared with 15.9 g. sucrose; the gels of the soft-wheat starch were slightly more rigid. With 23.7 g. sucrose, all of the starch gels decreased in rigidity and some retained their form for 30 to 60 seconds and then collapsed. With higher sucrose concentrations, pastes rather than gels resulted.

These results indicate a greater effect of sucrose than that reported by Woodruff and Nicoli (6), who found that 5% wheat and corn starch gels containing 10, 30, and 50% sucrose were well formed and that a syrupy mass resulted when 60% sucrose was used. These workers used small crucibles as gel molds, whereas the molded gels in this study were about 3 inches deep.

The soft-wheat flour gels became more rigid as the sucrose concentration was increased to 15.9 and 23.7 g. With 31.6 g. the gels retained their form for 30 to 60 seconds and then collapsed, and with the highest sucrose concentration a paste resulted. The hard-wheat flour gels showed a progressive increase in rigidity with each increase in sucrose. The effect of sucrose on the starch evidently is a minor factor determining the rigidity of flour-sucrose-water gels.

Water-Holding Capacity of the Gels. Data for the water-holding

capacity or syneresis of the aged starch and flour gels are shown in Table II. Syneresis of the starch gels increased with each increment of sucrose, indicating a lowered water-holding capacity. The corn-starch gels tended to show greater syneresis than the wheat-starch gels; the former also had lower solids, 6.5 g/100 g water, than the wheat starch gels, 7.3 and 7.5 g/100 g water for the hard- and soft-wheat starches.

TABLE II

SYNERESIS OF AGED GELS[a] OF STARCHES AND FLOURS WITH INCREASING SUCROSE CONCENTRATIONS

(Averages of three replicates)

THICKENING AGENT AND CONCENTRATION (Grams per 100 g. Water)	GRAMS SUCROSE PER 100 G. WATER				
	0.0	15.9	23.7	31.6	39.5
	Liquid from 50 g. of gel				
	g.	g.	g.	g.	g.
Corn starch 6.5	1.3	2.2	6.4	12.1	13.7
Hard-wheat starch . 7.3	0.3	2.6	5.4	9.1	10.9
Soft-wheat starch .. 7.5	0.4	0.5	3.8	8.6	11.1
Soft-wheat flour .. 11.3	0.0	0.0	0.0	0.8	3.3
Hard-wheat flour . 13.8	0.0	0.0	0.0	0.0	0.0

[a] Aged for 48 hours at 5°C.

Syneresis was not observed in any of the hard-wheat flour gels, but there was a slight amount in the soft-wheat flour gels made with the two highest concentrations of sucrose. The effect of sucrose on the starch is apparently a minor factor in determining the water-holding capacity of a flour; this parallels the observations on flour paste viscosity and gel rigidity.

Syneresis of the waxy starches could not be measured by this method because the pastes passed through the sieve.

Microscopic Measurements. The reliability of the estimates of the number of granules of different sizes and the volume of the swollen granules per g. of starch may be questioned for the following reasons: 1) sampling of the corn and wheat starches may not have been adequate because of the tendency of the hot starch pastes to aggregate when diluted with water at room temperature; 2) many of the largest granules may not have entered the counting chamber of the hemocytometer, because proportionately more large granules were observed on an ordinary slide than in the hemocytometer; 3) aggregates and granule fragments may have been counted as granules; 4) all of the swollen granules were not spherical as assumed. Recognizing these

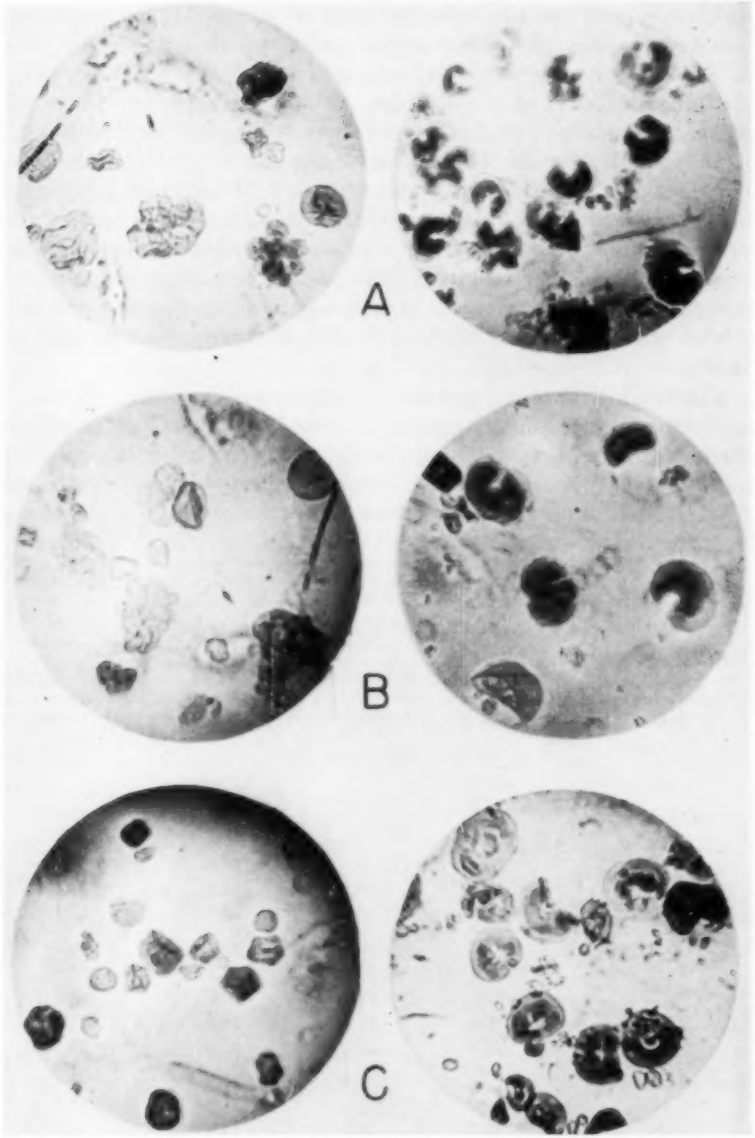

Fig. 3. Photomicrographs of corn starch (left) and wheat starch (right) heated to 95°C. (Magnification 208×.) A, heated in water alone; B, heated in 23.7 g. sucrose/100 g. water; C, heated in 39.5 g. sucrose/100 g. water.

limitations, only the trends of the effect of sucrose on the starch are discussed. Typical photomicrographs are shown in Fig. 3.

There was less disintegration and usually less swelling of the granules of each starch with increasing sucrose concentrations. No consistent trend was observed among the starches in the size distribution of the granules and in the volume of the granules per g. of starch. These may be a result of a greater estimated diameter of the plump and rounded granules of some of the starches in the solutions of the highest sucrose concentration, of a smaller estimated diameter of the wrinkled, folded, and saddle-shaped granules characteristic of some in the solutions of lower sucrose concentrations, and of the presence of more intact granules in the solutions of highest sucrose concentration.

Considering all of the starches, the effects of sucrose on the microscopic appearance of the granules, on the number of granules of different sizes, and on the estimated volumes occupied by the swollen granules lend support to the hypothesis that decreasing hydration and disintegration of the starch granules occur in the presence of increasing amounts of sucrose.

TABLE III

EFFECT OF SUCROSE ON THE QUANTITY AND COMPOSITION OF THE WATER SOLUBLE STARCH[a] OF CORN AND WHEAT STARCH
(Averages of three replicates)

STARCH	SUCROSE	SOLUBLE STARCH	IODINE AFFINITY OF SOLUBLE STARCH	LINEAR FRACTION IN SOLUBLE STARCH
	g/100 g water	%	%	%
Corn, 6.5 g.	0.0	12.4	16.6	88.9
	15.9	10.8	16.2	86.5
	23.7	10.6	16.8	89.9
	31.6	10.1	15.8	84.5
	39.5	8.0	16.7	89.3
Soft wheat, 7.5 g.	0.0	12.9	16.5	82.8
	15.9	11.1	17.2	86.4
	23.7	11.5	17.0	85.2
	31.6	10.8	18.5	91.0
	39.5	8.6	15.8	79.4
Hard wheat, 7.3 g.	0.0	9.8	16.4	82.2
	15.9	11.5	18.2	91.3
	23.7	10.4	16.8	84.6
	31.6	9.5	18.6	93.5
	39.5	9.4	17.8	89.5

[a] Calculated from the iodine affinity of the soluble starch; assuming 18.7% for pure corn amylose and 19.9% for pure wheat amylose.

Soluble Starch and Its Estimated Composition. The amount of soluble starch leached from the granules during heating, its iodine affinity, and the approximate amount of the linear starch fraction are shown in Table III. In general, the amount of soluble material decreased as the sucrose concentration increased. The yield of soluble starch from the water pastes of the hard-wheat starch was relatively small. These pastes did not pack well in centrifuging, and collection of all the supernatant was impossible.

The decrease in the amount of soluble starch followed the trends of lesser swelling of the granules and of decreased rigidity and increased syneresis of the corn and wheat starch gels prepared with corresponding concentrations of sucrose. The dissolved sugar molecules hydrate, leaving less free water for starch hydration. With decreased starch hydration, granule swelling would decrease with a subsequent reduction in the amount of solubles released from the granule. This could also account for decreased gel strength.

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A SEMIMICRO BAKING PROCEDURE, WITH MECHANICAL DOUGH HANDLING, PRIMARILY DESIGNED FOR EVALUATION OF ADDITIVES[1]

M. A. Cookson and Margaret L. Ritchie[2]

ABSTRACT

A method is described for test baking in which experimental error is reduced to a minimum by use of mechanical equipment (part of the Brabender Farinograph and Extensograph) for all dough-handling operations. In order that bread storage tests may be carried out in addition to the usual assessment of fresh loaf characteristics, three loaves are made from a bulk dough prepared with 250 g. flour. A convenient method of recording results in this test-baking procedure is given. A scheme for the incorporation of additives to doughs is described.

Although the "100-g. loaf" is the standard unit of test baking in the United States (1), and micro technics have been devised for quantities of flour from 10 g. to 25 g. (9), most of the test baking in Great Britain is done on 16-oz. or 32-oz. dough pieces. The microbaking methods reviewed by Shogren and Shellenberger (9) have been designed primarily to test flours of different baking strengths produced by micromilling small samples of wheats. For such purposes, the measurement of loaf volume and scoring of external loaf characteristics have been considered of primary importance. Both Geddes (6) and Harris (8) have pointed out that the surface of cut loaves prepared from 25 g. flour is too small to score satisfactorily for crumb color and texture, though Van Scoyk (10) appears to have obtained satisfactory internal as well as external comparisons on loaves of this size.

The procedure described in this paper was primarily designed for the evaluation of additives. For this purpose a reasonably accurate assessment of crumb firmness and texture as well as loaf volume is essential. Two hundred fifty grams of flour are used in a bulk fermentation, from which three 113-g. dough pieces are obtained. Thus, a set of three loaves is available for the measurement of loaf characteristics, and staling tests can be carried out on each batch. The process has been mechanized as much as possible to reduce operator variability in dough handling.

Materials and Methods

The test baking formula is based on a typical British straight-

[1] Manuscript received July 5, 1955.
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dough recipe for pan bread, and the technic differs from the standard A.A.C.C. method (1).

Formula.

Flour, 250 g.;
Yeast, 4.5 g. suspended in some of the dough water;
Salt, 3.5 g. as a 10% w/v solution;
Water, as determined by the water absorption figure for the flour, corrected for the water added as salt solution.

Addition of Additives. (a) Water-soluble additives were dissolved in part of the dough water. (b) Water-emulsifiable materials were added as emulsions to the dough after 1¼ minutes of mixing. (c) Shortenings and materials of a fatty nature were added after 1¼ minutes of mixing. (d) Oil-soluble materials which could not be incorporated by any of the above methods were dissolved in liquid paraffin (mineral oil) (2 ml.) and added after 1¼ minutes of mixing. (e) With defatted flour, oil-soluble additives which could not be added by either method (b) or method (c) were incorporated by dissolving in ether (50 ml.) and adding the resultant solution to defatted flour (50 g.). The slurry was transferred to a metal tray and left overnight at room temperature to allow the ether to evaporate, before the remaining flour was mixed in.

Dough Mixing. All doughs were mixed in the mixing unit of a Brabender Farinograph for 3.5 minutes. Additives were incorporated as described above. Confirmatory tests on the adequacy of mixing had shown that 2¼ minutes' mixing was sufficient for the complete homogenization of a colored oil phase into the dough. Similar results can be obtained using the single-blade mixing units fitted to the Simon Extensometer or the Chopin Alveograph, by increasing the mixing time to 6 and 7 minutes respectively. The mixing bowl was temperature-controlled to 27°C. (±0.5°C.) by means of a water jacket, but alternatively the mixing unit may be operated in a constant-temperature room maintained at the same temperature. The temperature of the dough water was adjusted to give a final dough temperature of 27°C. (±0.5°C.).

Fermentation. After mixing, doughs were placed in graduated glass jars (10 by 5 by 4 inches) fitted with screw caps. Thus, fermentation could take place in the relative humidity set up by the doughs without the necessity of a constant-humidity cabinet. The jars were kept in a constant-temperature room at 27°C. (±0.5°C.) or in a proofing cabinet maintained at the same temperature. To allow measurement of changes in dough volume during fermentation, the jars were

graduated linearly in terms of volume from bottom to top on two opposite sides.

Remixing. At the end of 180 minutes' fermentation, the dough volume and temperature were noted and the dough was removed from the fermentation jar to the original mixing bowl where it was remixed at slow speed for 20 seconds. During this remixing period the average consistency unit measured on the farinograph scale head was observed.

Recovery Period. After remixing, the dough was replaced in the fermentation jar which was again closed and left for 7 minutes at 27°C. (±0.5°C.) to allow the dough to recover from the severe remixing action before molding.

Molding. The bulk dough after recovery was divided and scaled into three pieces of 113 g. Each piece was molded on the ball molder and roller of the Brabender Extensometer. After 30 turns on the molder plate, the dough pieces were shaped on the roller, from which the back plate was removed to obtain dough pieces of suitable dimensions to fit the pans. Great care was taken during the dividing and molding stages to prevent formation of a "skin" on the dough surface. This was largely done by covering the dough pieces immediately after scaling with dome-shaped bowls measuring approximately 4 inches diameter by 1.5 inches high, and by carrying out the entire process in the constant-temperature room.

Proofing. The molded dough pieces were placed in bread pans constructed of tinned steel (standard wire gauge 27) of the following dimensions: 3.5 by 1.7 inches at the bottom, 4.1 by 2.5 inches at the top, and 2.0 inches deep. The pans were numbered 1, 2, 3, etc., according to the number of batches required, and each number coded a, b, and c for the triplicate dough pieces.

The replicate dough pieces from one bulk dough were placed in their appropriate coded pans on a shallow aluminum tray measuring 9.6 by 5.5 by 0.5 inch, and covered with an inverted normal 2-lb. baking pan of dimensions 7.4 by 4.0 inches at the bottom, 8.5 by 5.0 inches at the top, and 3.6 inches deep. The molded dough pieces were allowed to proof for 45 minutes at 27°C. (±0.5°C.). At the end of this period the inverted baking-pan covers were removed and proof heights were measured on a device designed by S. J. Cornford,[3] shown in Fig. 1. This consists of a counterbalanced pointer and scale mounted on a stand, the circular scale being graduated in 0.1-inch units.

Baking. The aluminum trays carrying the sets of three dough pieces in their pans were again covered with baking pans similar to

[3] Cornford, S. J. Unpublished work. British Baking Industries Research Association.

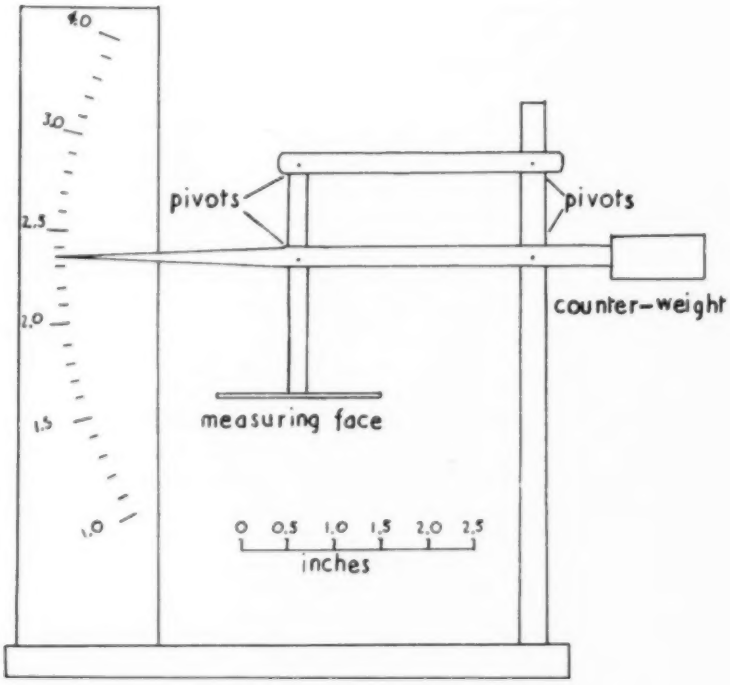

Fig. 1. Device for measuring proof heights of dough.

those described above, but having a series of five 1/8-inch perforations drilled at equal intervals along the longitudinal axis of the bases. A Simon glass-fronted electric oven measuring 11.5 inches wide by 20.5 inches deep by 11.5 inches high, fitted with an electric fan inserted through the roof to effect more even dispersion of heat and maintained at 230°C. (±5°C.), was used for baking. This oven was capable of baking six loaves arranged in two sets of three. After the first 10 minutes of the 20-minute baking period the perforated covers were removed, and baking continued normally. A series of doughs mixed at 10-minute intervals could thus be baked satisfactorily in this time schedule. Immediately after removal from the oven, the heights of the loaves (still in pans) were measured on the device shown in Fig. 1 and the loaves were removed from the pans and coded according to pan marking. With other types of ovens having more even heat dispersion, it might well be possible to bake without covering the doughs.

Measurement and Scoring. After cooling for 1 hour, the loaves were packed into three reasonably airtight containers (for the a, b,

and c loaves of different batches) and stored overnight at 21°C. About 18 hours after baking, the external characteristics and general replication were noted on all three loaves in each series. The "b" loaf from each batch was cut, and one half was ink-printed by running a black-inked rubber roller over the cut surface and impressing the loaf on absorbent paper supported on a felt pad. A qualitative assessment of crumb color and texture was made on the remaining cut surface. The other two loaves of the series were repacked into their containers and stored at 21°C. until required. About 24 hours after baking, crumb firmness was determined on two center slices of one of the loaves, using a compressimeter described by Cornford and Coppock (4). The third loaf of each series was stored at 21°C. for 4 days and then crumb firmness was again determined; thus the rate of increase of firmness of the bread was obtained. Volume was determined by seed displacement but could also be estimated from a correlation of waxed loaf volume with the cross-sectional area of the bread print determined with a fixed-arm planimeter. Grain can also be assessed from the ink print by comparing a cut-out section with a set of standard prints (cf. Harrell, 7).

Results and Discussion

Breads prepared from dough pieces weighing 16 oz. (453.6 g.) were compared with loaves made using the equipment and method described. The results indicated that the described technic was as capable of precision and replication as the standard 16-oz. process and had the advantage of using less material, which enabled the process to be carried out under laboratory conditions. Details of the technic were adopted for a variety of reasons:

The dough formula consisting of flour, yeast, salt, and water only was considered desirable for studies on *bread*. The sugar, milk powder, shortening, diastatic supplement, and yeast foods used in many formulas may falsify any results obtained unless the purpose of baking is to compare with an existing procedure, which would occur particularly in commercial test baking. In fundamental studies the other ingredients, owing to the wide variation in quantities used and their relative effects in bread, may give misleading results. However, there is no obvious reason why the procedure described in this paper should not be followed with other formulas or amended to resemble existing technics, e.g., by alteration of times and temperatures. The main feature of the procedure described is to have a test-baking system in which operative error or influence is reduced to a minimum.

The stated dimensions of fermentation jars and baking pans were

found convenient for the method described, but they can, of course, be altered provided they are of adequate volume for the size of dough.

Remixing the dough at the end of fermentation gave bread with a much finer texture and a noticeable absence of irregular gas holes, though a longer proof time (45 minutes) was necessary to obtain comparable proof heights. This short remixing period modified only to a very slight extent the typical characteristics of bread made from flours treated with such oxidizing improvers as potassium bromate (5). Thus breads made from undertreated, optimally treated, and overtreated flour were always readily distinguishable.

The observation of dough consistency on the farinograph scale head during remixing is intended mainly to indicate whether the doughs are of correct consistency, but changes might be anticipated with certain additives. A value of 410 ± 25 Brabender Units is average for this English dough formula. It would, of course, be likely to vary with other dough formulas.

It was found convenient to proof and bake the triplicate sets of dough pieces together. During proof, the 2-lb. baking pan placed over the three small pans resting on a lipped aluminum tray was sufficient to maintain a constant degree of humidity without the necessity of maintaining the trays in a constant-humidity cabinet.

Baking uncovered doughs always gave irregular oven-pull to a variable degree, depending upon the position of the loaf in the Simon oven. Covering the doughs with nonperforated covers during baking caused reduced oven spring and resulted in pale-colored, squat loaves characteristic of baking in the presence of excess steam. Using perforated covers throughout baking produced less obvious "steaming," but crust color was still very light, and the baking period was prolonged (35 minutes). Thus by removing the perforated cover after the dough had set in the oven, symmetrical loaves of good crust color were obtained.

In addition to the usual loaf characteristics (loaf weight and volume, crumb texture and firmness), some physical measurements of the dough – fermentation volume, dough consistency, and proof height – were also noted. These values, together with changes in the basic dough formula and other data, were recorded in tabular form (see Table I). This method of tabulation has proved concise, the results can be readily indexed, and the tabulated sheets can be conveniently stored in a looseleaf file with a facing sheet containing the appropriate bread prints (Fig. 2).

Despite attempts to minimize experimental error, slight differences in duplicate results have occurred, although these are much greater

TABLE I

EFFECT OF TEASEED OIL AT DIFFERENT WATER ABSORPTION LEVELS

Batch No.	I	II	III	IV
Type of flour	81% Extraction untreated flour			
Additives	None	2 ml. teaseed oil 2 lbs/sack	None	2 ml. teaseed oil 2 lbs/sack
Volume of water (ml.)	134	134	120	120
Dough Temp. (°C.):				
Initial bulk	80	80	80.5	80
Final bulk	82	82	82	82
Fermentation volume (ml.)	1040	1005	850	920
Resistance to remixing	450	400	600	600
Comments on preparation	These doughs have their correct water absorption		These doughs contain less water than I & II	
Proof height (in.)	2.25	2.20	2.20	2.25
Loaf height (in.)	3.10	3.10	3.10	3.25
Oven spring (in.)	0.85	0.90	0.90	1.00
Loaf volume (ml.)	355	350	350	375
Loaf texture index	3.0	2.5	1.0	1.5
Crumb color index	2.5	2.5	2.5	2.5
Crumb firmness (g.):				
24 hours	775	750	800	525
96 hours	1500	1500	1650	1250
Comments	Little significant difference		Considerable differences in main physical properties	

from day to day than within individual days. Possible sources of variance might include: 1) slight differences in batches of yeast: the volume of gas produced by consecutive batches of commercial pressed yeast has varied in a 4-hour fermentation by as much as 15%[4]; 2) changes in dough temperature and humidity from day to day; a ±1°C. difference from 27°C. in fermentation temperature has caused a difference of ±8% in volume of gas produced in a 4-hour fermentation[4]; and 3) a change in the water-absorption capacity of the flour; this increases erratically on storage more than can be accounted for by loss of moisture[4]. Although the increase amounts only to about ½ gal. per 280 lb. flour in 2 months, it was considered desirable, because of the erratic manner of the change, to determine the water-absorption capacity of the flour not more than 24 hours prior to test baking. It is difficult to see how, with this type of breadmaking system, the process

[4] Bennett, R. Unpublished work. British Baking Industries Research Association.

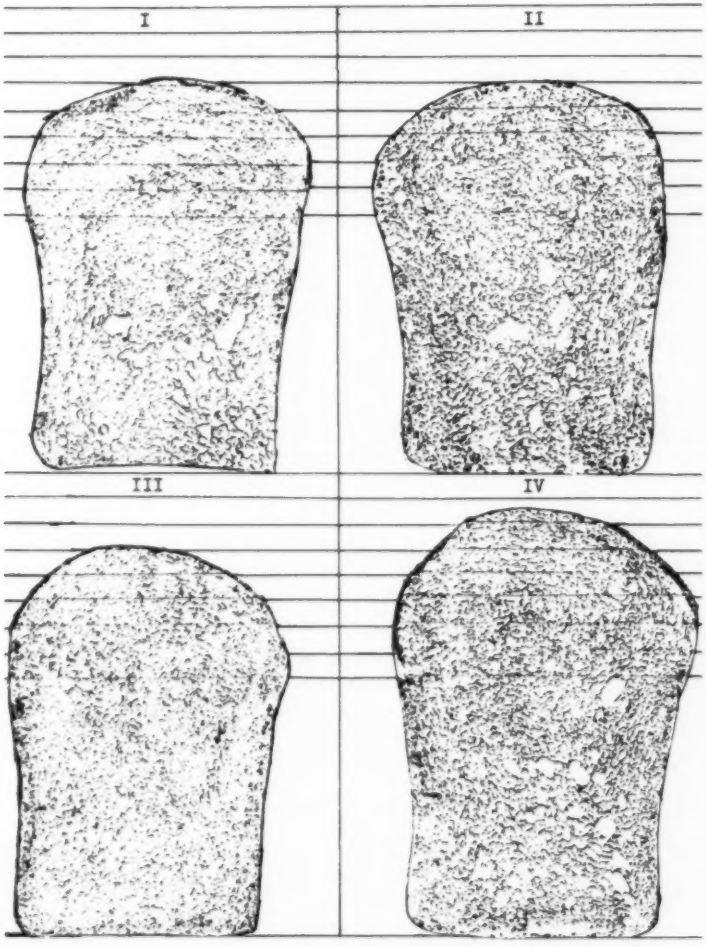

Fig. 2. Bread prints resulting from inking the cut surface with a rubber roller and pressing the loaf on absorbent paper supported by a felt pad.

could be better controlled further to reduce experimental error.

The addition of certain ingredients to the basic dough formula has been given careful consideration. Water-soluble additives are conveniently added in the dough water, and shortenings or other materials of a fatty nature are added as initially described. Water-emulsifiable materials (e.g. self-emulsifying glyceryl monostearate) can be added as emulsions in a manner similar to that with shortenings. Certain other additives, however, do not lend themselves to incorporation

by any of these methods. If such materials are oil-soluble, they may be added in a small amount (0.36% of the flour weight) of liquid paraffin (mineral oil) or vegetable oil, which has been found to be relatively inert in normal simple bread doughs. Nevertheless, the same amount of oil should also be added to a control dough. With defatted flour, on which much of the present work in these laboratories is concerned, it has not been found satisfactory to incorporate such additives in oil, owing to the depressing effect of oils on loaf volume.[5] In this case, the additive can be incorporated satisfactorily when a slurry of 50 g. of the defatted flour is made with a small volume (50 ml.) of ether containing the dissolved material and allowed to dry out before the remaining flour is mixed in. There is, of course, the possibility that an additive may still not be in a reactive form.

Further work is being carried out to determine whether dough characteristics can be measured on the Brabender Extensograph, and the dough pieces then further manipulated and baked. Thus, as much information as possible would be obtained from each batch of flour, dough, and bread.

Acknowledgments

The authors acknowledge their thanks to Dr. Coppock, Miss Bennett, and Messrs. Coupe, Devlin, Ottaway, and Price for their helpful advice.

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[5] It is generally agreed that in normal bread doughs, fats have an improving action on the resultant bread, while the usual edible oils have little or no improving effect (2, 3). With doughs containing less water (e.g. 1½ gal. per 280 lb. flour) than their optimum or correct water absorption value, this laboratory has found that oils are as effective as fats. This result is illustrated in Table I and Fig. 2.

A NEW METHOD OF FRACTIONATING WHEAT FLOUR[1]

WILLIAM F. SOLLARS[2]

ABSTRACT

Wheat flour has been fractionated by a method which does not depend on doughing and kneading under water. Flour was first extracted with water to obtain the water-solubles; the residue was then extracted with dilute acetic acid, and the extract was neutralized to recover the gluten. The final two-layer residue was separated into tailings and prime starch. Acid extractions at pH 2 were slightly more efficient, but extractions at pH 3 or higher resulted in better cookies from the reconstituted flours. A second acid extraction and the use of more than twice as much acid as flour were necessary for efficient separation. The yield and protein content of the fractions from the acid extraction process and the cookies baked from the flour reconstituted from these fractions compared favorably with similar results from hand and mechanically kneaded separations.

The acid extraction process required more time than separations made by kneading under water, hence is suggested for use with low-protein flours or those with damaged gluten.

All of the fractionation and reconstitution studies reported in the literature[3] have involved dough kneading and washing technics to separate the starch from the gluten. The exclusive use of this separation testifies to its efficiency and reliability. Most workers, though, have used high-protein, hard-wheat flours. Those who have worked with low-protein, soft-wheat flours have often reported difficulties in effecting good separations. This seems to be especially true for those using hand kneading (9, 13). However, machine kneading has been reported as successfully separating gluten from starch for any type of flour, even those with very weak gluten (4, 12). Probably personal factors enter into fractionation by kneading, some workers being more successful than others in separating gluten from low-protein flours.

In fractionation and reconstitution studies of cookie baking quality (11), the use of flours at 7 to 8% protein levels was desirable to conform to typical pastry flours of the Pacific Northwest. The method of fractionating these flours described here was developed some years ago and has been used since in locating the constituents responsible for cookie baking quality. The purpose of this paper is to report the de-

[1] Manuscript received September 19, 1955. Contribution from the Western Wheat Quality Laboratory, Department of Agricultural Chemistry, Washington Agricultural Experiment Stations, State College of Washington; and the Field Crops Research Branch, Agricultural Research Service, U. S. Department of Agriculture. Scientific Paper No. 1414, Washington Agricultural Experiment Station, Pullman.

[2] Western Wheat Quality Laboratory, Field Crops Research Branch, ARS, U. S. Department of Agriculture, and Department of Agricultural Chemistry, Agricultural Experiment Stations, Pullman, Washington.

[3] The work of Cunningham, Geddes, and Anderson on the preparation of proteins by acid extraction was reported after this paper was prepared. (Cunningham, D. K., Geddes, W. F., and Anderson, J. A. Preparation and chemical characteristics of the cohesive proteins of wheat, barley, rye, and oats. Cereal Chem. *32*: 91–107 (1955).)

velopment of this method and to compare it with conventional kneading separations.

General Considerations

A separation procedure should yield fractions as little altered from the native state as possible, and no essential constituents should be lost (2). Baking tests are the best measure of any damage to baking quality from fractionation (6). If reconstitution and baking are to follow fractionation, then the use of distilled water is to be preferred so that the water-solubles will not be contaminated. For cookie baking, avoidance of excessive kneading might be advantageous, since it has been recommended that the mixing action during the baking test be kept at a minimum (3).

Previous fractionations have yielded a water-soluble fraction, a gluten fraction with a protein content of 60 to 75%, and a crude starch fraction with a protein content under 1%. It was conceived that these same fractions could be obtained by first extracting the material soluble in water, then extracting the gluten from the remaining dough with a protein solvent affording easy recovery of undamaged gluten, leaving a final residue of crude starch. The effectiveness of separating the gluten from the starch could be measured by protein analysis, while any damage during fractionation would be shown by the baking test. An extraction process such as this would yield fractions similar to those obtained by kneading procedures yet would avoid the objectionable features of the latter.

The necessity of recovering the gluten in a relatively unaltered form for use in reconstitution and baking limits the choice of protein solvents. In bread baking tests, Lusena (6) found that acetic acid dispersion and recovery caused no damage to gluten if the pH was 5.0 or above. Dilute acetic acid was reported by Sandstedt and Blish (10) to afford good dispersion and easy recovery with gluten, but when it was used with flour, gluten dispersion was incomplete. Mangels and Martin (7) determined the amount of total nitrogen dispersed by a number of acids and obtained as their highest yield 85.2% of the total nitrogen peptized by 0.5 *N* acetic acid. Lusena (6) showed that the water-soluble ash of flour greatly reduced the solubility of gluten in acetic acid, but that starch did not. Since the soluble ash could be removed by a preliminary water extraction, it appeared feasible to extract the gluten with acetic acid.

At the beginning, flour was separated into only the three abovementioned fractions; consequently, most of the work reported here was designed to yield only these three fractions. After the importance

of the crude starch fraction to cookie quality was discovered, this fraction was then separated into the tailings fraction and the prime starch. The final results and recommended procedure of this study deal with four fractions.

Materials and Methods

Straight-grade, unbleached flours milled on the Buhler mill from pure varieties were used. These varieties represented club, soft white, and hard red winter types ranging in protein content from 4.7 to 15.8%. Moisture, ash, and protein analyses and pH determinations were made by conventional methods (1, 5). The apparatus and procedures for shelling and lyophilization were similar to those described by Yamazaki (13, 14). The reconstitution procedure also was essentially that devised by Yamazaki (13, 14); however, the conditions, particularly the amount of water and the mixing time during the reconstitution doughing, were adjusted so as to yield cookies with diameters as close as possible to those of the cookies from the original flour.

Results

Effect of pH Level on Acid Extraction. Preliminary extractions with 0.005 *N* acetic acid gave poor separations; therefore, the effect of pH on the amount of protein extracted was investigated. The water-solubles were extracted from 200 g. of flour by stirring for 10 minutes with 500 ml. of distilled water, centrifuging for 10 minutes at 1900 r.c.f., and decanting. The residue or dough was transferred to a Waring Blendor and suspended for 1 minute in 400 ml. of distilled water to which various amounts of acetic acid and distilled water were added to obtain the desired pH and a total volume of 500 ml. For the lowest pH level, the doughs were suspended in glacial acetic acid only.

After 1 minute's agitation in the blender, each acid suspension was centrifuged for 10 minutes at 1900 r.c.f. The acid extract was then decanted. The residue or crude starch was resuspended in a minimum of water, the liquid removed by filtration in a Büchner funnel, and the moist cake removed, cut up, and air-dried.

The proportion of protein in each fraction extracted at the different pH levels is shown for two varieties in Table I. Similar results were obtained with other varieties. At pH levels of 2 to 3, from 70 to 77% of the total protein was contained in the acid extract, but the protein content of the crude starches still exceeded 1%. Preliminary reconstitutions and baking tests yielded good cookies from fractions separated at pH 3, but decidedly poorer cookies from those separated at pH 2. Fractionation at pH 3 was followed in the rest of this study, and efforts

TABLE I

DISTRIBUTION OF FLOUR PROTEIN AMONG FRACTIONS AND PROTEIN CONTENT OF CRUDE STARCHES AT VARIOUS pH LEVELS

VARIETY AND FLOUR PROTEIN	pH of ACID EXTRACTION	PROPORTION OF FLOUR PROTEIN IN:			TOTAL	PROTEIN CONTENT OF CRUDE STARCH[a]
		Water Extract	Acid Extract	Crude Starch		
		%	%	%	%	%
Rio, 8.1%	5.0	8.8	26.2	64.5	99.5	5.81
	4.0	8.8	62.5	26.4	97.7	2.48
	3.0	8.9	70.6	16.7	96.2	1.58
	2.0	8.8	76.6	12.3	97.7	1.16
	1.1	8.8	66.6	22.9	98.3	2.11
Elgin, 8.2%	5.0	9.7	60.1	27.9	97.7	2.61
	4.0	9.8	72.1	17.6	99.5	1.64
	3.0	9.4	77.3	11.5	98.2	1.07
	2.0	10.4	74.9	11.8	97.1	1.09
	0.9	9.9	67.5	19.9	97.3	1.80

[a] At 14% moisture.

TABLE II

DISTRIBUTION AND RECOVERY OF FLOUR PROTEIN AMONG FRACTIONS WITH TWO ACID EXTRACTIONS

FRACTION	DISTRIBUTION OF FLOUR PROTEIN							
	Rio, 8.1% Protein				Elgin, 8.2% Protein			
	Liquid Extracts		Dry Fractions		Liquid Extracts		Dry Fractions	
	%	%	%	%	%	%	%	%
Water-solubles		8.2		8.2[a]		9.3		9.0[a]
Acid-ext. 1	68.8		55.8		71.6		65.1	
Acid-ext. 2	12.8		7.7		11.6		9.0	
Total acid-ext.		81.6		63.5		83.2		74.1
Crude starch		(8.3)[d]		8.3		(5.3)[d]		5.3
Total protein recovery		98.1		80.0		97.8		88.4
Starch filtrate[b]		1.4				1.1		
Protein content of crude starch[c]				0.80				0.51

[a] Concentrated under vacuum below 40°C., shelled, and lyophilized.
[b] Discarded filtrate from crude starch suspension.
[c] At 14% moisture.
[d] Protein of dried starch included here to calculate total recovery.

were made to increase the amount of protein extracted at that level.

Effectiveness of a Second Acid Extraction. Fractionations were made in which a second acid extraction was performed immediately following the first, again using 500 ml. of acid at pH 3.0. The second

suspension was then treated as before to recover a second acid extract and an improved crude starch. The filtrate from the starch suspension was saved for protein determination. The acid extracts were neutralized with 5 *N* sodium hydroxide to pH 6.4 and centrifuged for 20 minutes. The gluten residue was cut into thin strips and air-dried.

The results (Table II) show that the second acid extraction effected an increase of over 10% in the amount of flour protein appearing in the acid extracts and yielded crude starch fractions with protein contents well below 1%. When the dry fractions were obtained, however, a considerable loss of protein occurred through failure of a portion of the gluten to precipitate.

Ratio of Acid Solution to Amount of Flour. This variable was studied to determine if a reduction of the volume of supernatant after neutralization would increase the amount of protein recovered. Flour of 6.9% protein content from Elgin wheat, after removal of the water-solubles, was extracted twice at pH 3.0 as before, except that the volume of acid was altered from the previous proportion of 500 ml. to the starting weight of 200 g. flour. The fractions were recovered, flours were reconstituted from each fractionation, and cookies were baked from the flours.

The results (Table III) show that the first acid extraction, with ratios of acid to flour of 2 to 1 and lower, gave the poorest recoveries of gluten. For the second acid extraction, ratios between 1 to 1 and 2 to 1 appeared satisfactory. All fractionations yielded cookies of about the same quality. The ash contents of the flours reconstituted from the fractionations at the lower ratios were excessive, but those of the flours made up from the fractionations at ratios of more than 2 to 1 were nearly identical with the ash content of the original flour.

TABLE III

DISTRIBUTION OF FLOUR PROTEIN IN THE DRY FRACTIONS OBTAINED WITH VARIOUS ACID-FLOUR RATIOS

RATIO, ML. OF ACID TO G. OF FLOUR	PROPORTION OF FLOUR PROTEIN					ASH CONTENT[a]
	Water-Solubles	Extract 1	Extract 2	Crude Starch	Total	
	%	%	%	%	%	%
0.75:1.0	10.2	49.6	9.4	4.4	73.6	...
1.0 :1.0	10.6	51.6	9.2	5.6	77.0	1.58
1.5 :1.0	10.6	54.7	9.3	5.7	80.3	1.68
2.0 :1.0	10.7	58.2	8.8	5.5	83.2	0.50
2.5 :1.0	11.6	61.7	7.3	5.0	85.6	0.47
3.0 :1.0	11.5	62.0	4.7	6.8	85.0	0.38
4.0 :1.0	11.2	61.7	3.7	4.9	81.5	0.38

[a] Ash content of flours reconstituted from these fractions.

Direct lyophilization of the acid dispersion was accomplished several times. The recovery of gluten was considerably improved, but the reconstituted flours containing the gluten obtained by this method yielded cookies of poor quality.

Effect of Temperature. Fractionations were made of Elgin flour at 27° and at 1°C. by adding cracked ice made from distilled water to the latter lot to control the temperature. Very little difference was apparent, in the yield or protein content of the fractions, and the cookies baked from the flour reconstituted from the fractions separated at 1°C. showed only slight improvement.

In another fractionation, the acid dispersion was neutralized and then heated to 96°C. for 5 minutes to inactivate any remaining enzymes (8). The dispersion was cooled and the gluten recovered. The cookies baked from flours containing this gluten were somewhat poorer in quality.

Comparison with Conventional Kneading Separations. Flour of 8.0% protein content from the Golden variety was fractionated by three methods: (a) by hand kneading according to a procedure reported to give good separations with soft-wheat flours (13); (b) by a mechanical kneading procedure (12); and (c) by the acetic acid fractionation scheme given later in this report. The fractions were recovered, reconstituted flours prepared, and cookies baked. The yield and protein content of the fractions are listed in Table IV; the cookies are shown in the top row of Fig. 1.

The gluten and the water-solubles from the mechanical kneading procedure were similar in yield and protein content to the gluten and the water-solubles from the acid-extraction process. However, in the

TABLE IV

A Comparison of Three Methods of Flour Fractionation

Fraction	Hand-Kneaded		Mechanically Kneaded		Acetic Acid Extracted	
	Yield[a]	Protein[b]	Yield	Protein	Yield	Protein
	g.	%	g.	%	g.	%
Water-solubles	6.1	19.0	4.8	15.3	4.5	15.9
Gluten	12.6	40.4	9.9	64.3	8.6	66.4
Tailings	46.9	2.30	35.1	1.10	21.9	2.03
Prime starch	31.2	0.64	45.4	0.23	61.1	0.31
Dry matter recovery[a]		96.8		95.2		96.1
Protein recovery		94.0		94.8		88.1

[a] Grams at 14% moisture from 100 g. of flour at 14% moisture.
[b] At 14% moisture.

acid-extraction process, the tailings fraction was more compact with sharper separation from the prime starch; consequently, the tailings fraction from this process was smaller and higher in protein than that from the mechanical kneading. The compact tailings also resulted in a higher yield of prime starch from acid extraction.

In hand kneading, it was very difficult to prevent disintegration of the dough. The gluten fraction was low in protein, and the tailings layer was poorly defined. The hand-kneading procedure has consistently yielded a water-soluble fraction higher in both yield and protein content than the water-soluble fraction from the acid-extraction process.

The flours reconstituted from the fractions of the two kneading procedures gave cookies of similar appearance. Underdevelopment of the top grain seems to be characteristic of kneading separations (13, 14). The flours reconstituted from fractions of the acid-extraction method gave cookies with somewhat overdeveloped top grain, the valleys being deeper and the islands being fewer in number and larger than the ones in cookies from normal flours. Color of all cookies from reconstituted flours was similar.

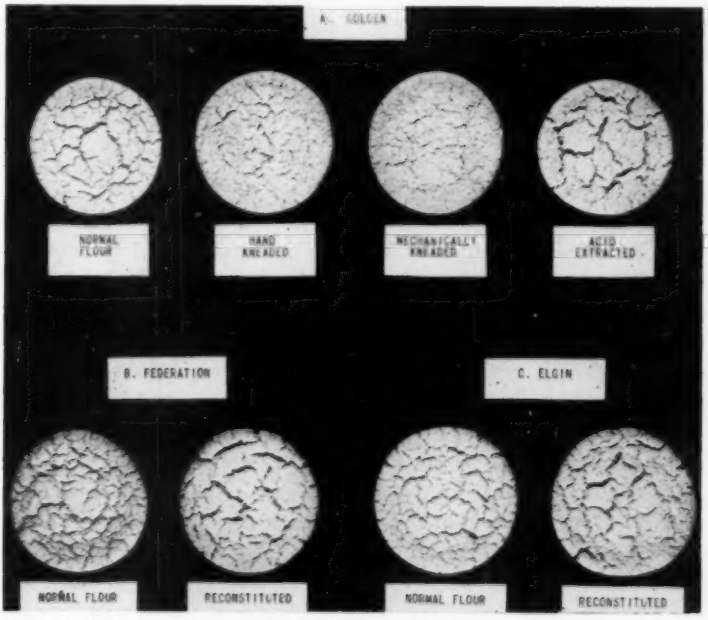

Fig. 1. Cookies from normal and reconstituted flours: A, Golden, fractionated by three methods; B and C, Federation and Elgin, fractionated by the acid-extraction method.

TABLE V

MATERIALS BALANCES OF TYPICAL ACID FRACTIONATIONS

VARIETY AND FLOUR PROTEIN	FRACTION	YIELD[a]	PROTEIN CONTENT[b]	AMOUNT OF PROTEIN	RECOVERIES	
					Materials	Protein
%		g.	%	g.	%	%
Elmar, 4.7%	Water-solubles	3.9	17.0	0.66		
	Gluten	5.0	61.2	3.06		
	Tailings	26.3	0.88	0.23		
	Prime starch	62.3	0.20	0.12	97.5	86.6
Elgin, 6.9%	Water-solubles	4.4	16.7	0.74		
	Gluten	7.2	65.0	4.68		
	Tailings	17.0	1.43	0.24		
	Prime starch	69.4	0.20	0.14	98.0	84.1
Rio, 6.8%	Water-solubles	5.1	12.8	0.65		
	Gluten	6.7	66.1	4.43		
	Tailings	20.4	2.15	0.44		
	Prime starch	64.4	0.22	0.14	96.6	83.2
Golden, 8.0%	Water-solubles	4.4	15.9	0.70		
	Gluten	8.8	64.5	5.68		
	Tailings	21.9	1.77	0.39		
	Prime starch	62.5	0.23	0.14	97.6	86.4
Triumph, 15.8%	Water-solubles	4.3	21.8	0.94		
	Gluten	18.1	72.3	13.25		
	Tailings	19.8	2.74	0.54		
	Prime starch	56.5	0.26	0.15	98.7	94.2

[a] Grams at 14% moisture from 100 g. flour at 14% moisture.
[b] At 14% moisture.

Cookies from other normal and reconstituted flours are shown in the bottom row of Fig. 1 to demonstrate that other varieties can be fractionated by this method.

Application to Flours of Varying Protein Content. A number of flours with protein contents of 4.7 to 15.8% were fractionated by the acetic acid process. A materials balance for each of these fractionations is given in Table V. It is evident that this process is adaptable to a wide range of protein levels.

Discussion

That flour fractionation can be accomplished without dough kneading has been shown. By first extracting the flour with distilled water followed by dilute acetic acid, fractions were obtained that were similar to those produced by conventional kneading procedures. The pH

appears to be the most important factor in acid extractions. A pH of 4 or lower was necessary for efficient separations, but poor reconstituted cookies resulted from using a pH lower than 3. Hence pH levels between 3 and 4 were the most useful. A pH of 4 could have been used with only a slight loss of efficiency and probably would have resulted in cookies of higher quality.

The acetic acid fractionation required more time than the kneading procedures and, therefore, probably is best adapted for use with low protein flours or those with damaged or altered gluten. The acid separation yielded a well-defined, compact tailings layer. The water-soluble fraction was removed early in the process and was quickly cooled or lyophilized. Any damage to the remaining fractions through exposure to dilute acetic acid for short periods of time must have been slight, as shown by the quality of the cookies baked from the reconstituted flours. Recovery of gluten was lower than desired, but the gluten lost was not important to cookie quality. Fractionation was satisfactory at a wide range of temperatures.

The fractionation of low-protein (8% or less) flour from Rio wheat, a hard red winter variety, by any of the three methods was more difficult than the fractionation of low-protein, soft-wheat flours. With kneading methods, the dough from Rio flours was particularly lacking in cohesiveness and it disintegrated easily. In acid fractionations, either the crude starch or the tailings fraction, when separated, was higher in protein than similar fractions from soft-wheat flours.

Recommended Procedure for Acetic Acid Fractionation of Flour

Water Extraction. Weigh 200 g. flour into 500 ml. distilled water and stir for 10 minutes with a propeller-type laboratory stirrer. Centrifuge the suspension for 10 minutes at 1900 r.c.f. Decant the supernatant, concentrate it under vacuum below 40°C. to 200 ml., shell, and lyophilize. Store in a tight container below 0°C.

Acid Extraction. Transfer the dough from the centrifuge bottles to a Waring Blendor cup and add 500 ml. acetic acid solution of sufficient strength (usually 25 ml. glacial acetic acid and 475 ml. distilled water) to produce a pH of 3.0. Agitate for 1 minute, centrifuge for 10 minutes, and decant the acid extract into a beaker. Again extract the residue for 1 minute with acetic acid solution of sufficient strength (usually 5 ml. glacial acetic acid and 400 ml. distilled water) to maintain the pH at 3.0, centrifuge, and decant.

Recovery of Gluten. Neutralize the combined acid extracts with 5 *N* sodium hydroxide to pH 6.4. Centrifuge the neutralized extracts

for 30 minutes at 1900 r.c.f. Remove the gluten residue and cut it into thin strips. Either air-dry or lyophilize the gluten strips.

Recovery of Tailings and Prime Starch. Remove the upper layer of the residue in the centrifuge bottles with a spatula, and either air-dry or suspend it in water, shell, and lyophilize. Suspend the lower, white, prime starch layer in water, remove the liquid by filtration in a Büchner funnel, cut the moist cake into thin strips, and air-dry or suspend the moist cake in water, shell, and lyophilize.

Storage. The water-solubles should be kept below 0°C. The dried gluten, tailings, and prime starch can be stored in tight containers at room temperatures for several days without damage to baking quality.

Acknowledgment

The author is indebted to Dr. W. T. Yamazaki for supplying the details of his fractionation and reconstitution procedures at the beginning of this study.

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EVALUATION OF FLOUR FRACTIONS FOR THEIR IMPORTANCE TO COOKIE QUALITY[1]

WILLIAM F. SOLLARS[2]

ABSTRACT

Wheat flours of different cookie baking quality were separated into water-solubles, gluten, tailings, and prime starch by an acetic acid fractionation process. These fractions were reconstituted into a flour suitable for baking cookies by a procedure such that the cookies obtained matched closely in diameter the cookies obtained from the original flour of the same variety. One fraction at a time was then interchanged with similar fractions from varieties differing in pastry quality.

At the same absorption level, blends of soft-wheat fractions took longer to mix to optimum dough development than did blends of hard-wheat fractions. For those blends with one interchanged fraction, the tailings affected the time of optimum development greatly, and the prime starch slightly.

The tailings fraction had a far greater effect on cookie diameter than the other fractions and appears to be the most important one in cookie quality. The water-solubles had a small but consistent effect on diameter; in view of the small amount of this fraction, the effect was relatively large per gram of material. The prime starch had a slight effect on diameter. The importance of gluten, although minor, was difficult to determine since the response from this fraction was erratic.

That reconstituted or synthetic flours have unique advantages in the study of wheat flour quality has been pointed out by several workers. Finney (3) used such flours to locate the constituents responsible for baking quality of bread and to determine the importance of the water-soluble fraction. Pence, Elder, and Mecham (6) prepared a number of such flours to study the effects of soluble components on baking behavior of bread. Bechtel and Meisner (2) and Prentice, Cuendet, and Geddes (7) resorted to their use to study the effect of each fraction on bread staling without introducing variations from other sources. These workers and others mentioned by them have used such flours to study baking quality of bread.

Yamazaki (9) appears to have been the first to extend the use of reconstituted flours to the study of soft-wheat quality. He investigated many variables of both fractionation and reconstitution, and developed a successful reconstitution method for cookie flours. More recently Yamazaki (10) reported further progress in concentrating a factor responsible for cookie quality. The hydration characteristics of

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the gluten, prime starch, and starch tailings fractions were determined. The factor located in the tailings was then concentrated, using hydration capacity as a criterion of cookie quality. Reconstituted flours were used only to check the results obtained by hydration tests. However, when such flours were used, evidence indicated that other factors besides the tailings were important to cookie quality.

The importance of the cookie baking test to soft-wheat quality evaluation appeared to justify further investigation. In this study all four fractions were assessed for importance to cookie quality, including the water-solubles which cannot be evaluated by hydration tests. The importance of each fraction was determined by the technic of reconstitution and interchange of corresponding fractions one at a time. The fractions, obtained by the acid fractionation method previously described (8), were more sharply differentiated from one another than were those obtained by hand kneading of low protein flours. This paper reports the evaluation of the four fractions.

Materials and Methods

Typical pastry flours of the Pacific Northwest are made from wheat of 8 to 9% protein. Pure varieties of hard and soft wheats of this protein level were milled on the Buhler mill to yield straight-grade, unbleached flours of 7 to 8% protein.

Moisture and protein content of fractions and flours were determined by conventional methods (1, 5). Flours were fractionated by the acetic acid process reported by Sollars (8). Apparatus and procedures for shelling and lyophilizing were similar to those described by Yamazaki (9, 10).

The reconstitution method developed by Yamazaki (9, 10) was used as the basic process, and modifications were made in it. The major steps of this process were making a blend of the dry fractions, adding water and mixing to form a dough, lyophilizing, grinding, and rehydrating to normal moisture levels. Protein contents of the reconstituted flours were determined frequently to ensure against appreciable changes.

Cookies were baked from normal and reconstituted flours by the micro baking procedure of Finney, Morris, and Yamazaki (4). The average diameter of two cookies was taken as the indication of cookie quality.

The method used to locate the fractions important to cookie quality was similar to that used by Finney (3) and others. In the first portion of this study two wheat varieties were chosen to represent the extremes of soft-wheat quality. Rio wheat flour was taken as an example of a

hard-wheat type that yields cookies of small diameter with poor top grain, properties associated with poor soft-wheat quality. Elgin, a club-wheat variety, was taken as the example of good soft-wheat quality; it yields cookies of large diameter and pleasing top grain. Typical yields and analyses for these fractions have been reported elsewhere (8). In evaluating the fractions, blends were made up of one fraction of one variety with the three remaining fractions of the other variety, and cookies were baked from the reconstituted flour. By comparison with the cookies baked from the original reconstituted flour, the effect of each substituted fraction was determined.

Later the study was extended to two more varieties. Golden, a soft common wheat, was chosen as another example of good soft-wheat quality; Wasatch, a hard red winter wheat, was taken as another example of poor soft-wheat quality.

Results

In the preliminary reconstitutions and interchanges of only the three fractions, crude starch, gluten, and water-solubles, the cookies often did not match the diameter of the check cookies from the original, nonfractionated flours. In spite of these irregularities, when one fraction at a time was interchanged in these early reconstitutions, it was evident that the crude-starch fraction was exerting a large effect on cookie diameter. This fraction was therefore divided into the tailings and the prime starch in subsequent fractionations, and the reconstitution procedure was studied to determine if it could be improved to yield cookie diameters that would match those for the original flours.

Experiments in making up flours with various combinations of lyophilized and air-dried fractions showed that all air-dried fractions, except for the water-solubles, were equal to or slightly better than lyophilized fractions in cookie baking tests. All fractions, except for the water-solubles, were thereafter prepared by air-drying. When the water-solubles from the original extract were lyophilized, an inconveniently fluffy, light-weight product was obtained. Concentration of the extract before lyophilization (6) resulted in a more compact and desirable product. Cookies baked from reconstitutions made with concentrated and lyophilized solubles showed that the solubles were unaltered by this process.

A deterioration in characteristics with time of storage was indicated when quantities of fractions were stockpiled in an attempt to achieve a uniform supply for reconstitution. Succeeding reconstitutions from the same fractions gave cookies with increasing deviations from the

diameter of the checks as compared to the first reconstitution. Thereafter reconstitutions were made as quickly as possible after fractionation.

Uniform treatment could not be maintained for all blends during reconstitution. Rio flour has a high optimum absorption of about 68 to 70% and a long mixing time, whereas Elgin flour has a low optimum absorption of about 56 to 58% and a short mixing time. Blends made up of fractions all from one variety had absorptions and mixing times similar to those of the original flour. With uniform treatment during the reconstitution doughing, one of the blends of all fractions from the same variety invariably resulted in doughs that were not at optimum development, thereby yielding cookies of abnormal diameters compared with those for the checks or original flours.

The procedure finally adopted was to allow one variable factor during mixing and to regulate it to produce optimum dough development. The major variables during mixing were the absorption level and time. Time was allowed to vary, since mixing could be conveniently stopped at the time of optimum dough development for each blend. The same amount of water (65%) was added to all blends. This amount of water was relatively high for blends of all Elgin fractions, and consequently a long mixing time was required to reach optimum development. Blends of Rio fractions, because of their high water absorption, required only a short mixing time.

A distinct change in the time to reach optimum dough development occurred when interchanging the tailings fraction and, to a smaller extent, the prime starch. At 65% absorption, blends of all Rio fractions formed a dough in 10 to 15 seconds of mixing and reached optimum development in about 3 minutes. A similar behavior occurred in the blends when Elgin water-solubles or gluten were substituted for the corresponding Rio fractions. Rio fractions plus Elgin tailings, however, behaved in a manner almost identical to that of the blend made up of all Elgin fractions. These two blends took a full minute to form a dough and reached optimum development only after 6 to 7 minutes. A summary of this behavior follows:

	Optimum development, in minutes		
Rio reconstituted flours		*Elgin reconstituted flours*	
All Rio	3	All Elgin	7
Elgin solubles	3	Rio solubles	7
Elgin gluten	3	Rio gluten	8
Elgin tailings	7	Rio tailings	3
Elgin prime starch	5	Rio prime starch	5

A rough estimation of the cookie diameter from each blend could be

predicted at the time of mixing by the mixing behavior.

The reconstitution method for cookie flours as developed by Yamazaki (9) thus was modified by using the acetic acid process for fractionation, followed by air-drying the gluten, tailings, and prime starch, by concentrating and lyophilizing the water-solubles, by reconstituting as soon and as rapidly as possible after fractionating, and by mixing at a uniform 65% absorption level and to optimum development by means of a variable mixing time. It was then possible to obtain reconstituted flours that yielded cookies of satisfactory top grain and with diameters that closely matched those for cookies from the original, nonfractionated flours.

The average diameters of the cookies from four complete, independent series of fractionations and reconstitutions in which one fraction at a time was interchanged between blends of Rio and Elgin fractions are shown in Table I.

TABLE I

DIAMETERS OF COOKIES FROM FLOURS OF GOOD AND POOR VARIETIES

TREATMENT	RIO [a]	ELGIN [a]	WASATCH [b]	ELGIN [b]	RIO [b]	GOLDEN [b]	WASATCH [b]	GOLDEN [b]
	cm.	*cm.*	*cm.*	*cm.*	*cm.*	*cm.*	*cm.*	*cm.*
Original flours	8.16	9.07	7.75	8.90	8.09	8.75	7.46	8.60
Reconstituted:								
All one variety	8.20	9.11	7.78	8.86	7.92	8.70	7.82	8.77
Interchanged solubles	8.31	8.85	7.83	8.64	8.23	8.21	7.98	8.61
Interchanged gluten	8.18	8.96	7.74	8.39	7.70	8.64	7.65	8.48
Interchanged tailings	8.73	8.64	8.11	8.43	8.27	8.20	8.03	8.20
Interchanged prime starch	8.44	8.97	7.84	8.48	7.93	8.52	7.78	8.49

[a] Average diameters for four independent series of reconstitutions in which one fraction at a time was interchanged between the two varieties shown. The standard error of a mean was 0.082 cm.

[b] Diameters for a single series of reconstitutions in which one fraction at a time was interchanged between the two varieties shown. The standard error of observation was 0.163 cm.

The cookies from one such series are shown in Fig. 1. All of each series were baked on the same day from flours made up with the fractions from two separations, one with Rio and one with Elgin flour.

One fraction at a time was then interchanged for only one series of reconstitutions involving an interchange of fractions of the following pairs: Wasatch and Elgin; Rio and Golden; and Wasatch and Golden. In these studies the investigation of optimum conditions for individual blends was limited. The results given in the last three sections of Table I represent somewhat poorer agreement in cookie quality between reconstituted and normal flours of the same variety.

Interchanging of the fractions one at a time was made between two hard-wheat and two soft-wheat blends to test the validity of the recon-

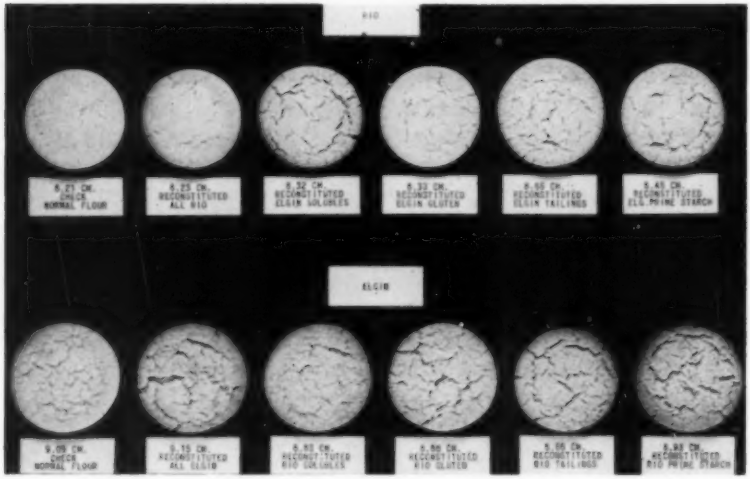

Fig. 1. Cookies from a typical reconstitution in which one fraction at a time was interchanged between varieties having very different soft-wheat quality.

TABLE II

DIAMETER OF COOKIES IN WHICH ONE FRACTION AT A TIME WAS INTERCHANGED BETWEEN TWO VARIETIES OF SIMILAR QUALITY

TREATMENT	RIO	WASATCH	ELGIN	GOLDEN
	cm.	*cm.*	*cm.*	*cm.*
Original flours	8.19	7.81	9.04	8.68
Reconstituted:				
All one variety	7.98	7.64	8.91	8.49
Interchanged solubles	8.02	7.68	8.91	8.61
Interchanged gluten	7.89	7.76	9.16	8.53
Interchanged tailings	7.83	7.71	8.93	8.46
Interchanged prime starch	7.67	7.70	8.76	8.89

stitution procedure. If interactions were occurring between the introduced fraction and the three original fractions, cookies with diameters outside the narrow range for two similar varieties would be expected to appear. The results (Table II) show that the diameters were about as expected for the interchanges between two similar varieties.

Discussion

The technic of fractionating and reconstituting flours appears to offer a reliable way of evaluating flour fractions for their effect on cookie quality. The close agreement in diameter between cookies baked from reconstituted flours containing the fractions from one variety and

those from the original flour of the same variety has been shown. It was also evident that each of the four fractions affected the differences between cookies baked from hard- and soft-wheat flours. The difference in cookie diameter between all Rio reconstituted flours and all Elgin reconstituted flours was 0.91 cm. The effect on diameter and the weight of each fraction are given in Table III.

TABLE III

EFFECT ON COOKIE DIAMETER FROM INTERCHANGE OF FRACTIONS AND WEIGHT OF EACH FRACTION IN 40 GRAMS OF FLOUR

Fraction	Weight of Fraction, Dry Basis		Effect on Cookie Diameter	
	Elgin	Rio	Rio	Elgin
	g.	*g.*	*cm.*	*cm.*
Water-solubles	1.5	1.7	0.11	−0.26
Gluten	2.9	3.0	−0.02	−0.15
Tailings	6.8	6.5	0.53	−0.47
Prime starch	23.2	23.2	0.24	−0.14
Total	34.4	34.4	+0.86	−1.02

From the results given above and others listed in Table I, it appears that each flour fraction is contributing to cookie quality. In most of the reconstitutions, however, the tailings fraction exerted a far greater effect on diameter than did any of the other fractions, and therefore it must be considered the most important fraction in cookie quality. This agrees with the report of Yamazaki.

The water-soluble fraction had a small but consistent effect on cookie diameter, particularly when the small quantity of this fraction is considered. The Rio water-solubles, in particular, exerted a relatively large effect per g. of material. The effect of the prime starch also was small and was emphasized by the large quantity of this fraction.

As shown in Fig. 1, the reconstituted Rio cookie with Elgin water-solubles had a top grain similar to that of the reconstituted all-Elgin cookie. Likewise, the reconstituted Elgin cookie with Rio water-solubles had a top grain similar to that of the reconstituted all-Rio cookie. This effect on top grain and the large effect per g. on diameter indicate that a study of the water-soluble fraction might be a useful approach to the problem of cookie quality.

Gluten was the most difficult fraction to assess for its contribution to cookie quality. The diameter response from this fraction was very erratic, sometimes large and at other times negligible. Because of the occasionally large responses and the small weight of this fraction, it

cannot as yet be dismissed as unimportant to cookie quality. Further study is needed to clarify its role.

Acknowledgment

The courtesy of Dr. William T. Yamazaki in providing the details of his fractionation and reconstitution procedures at the beginning of this study is gratefully acknowledged.

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ENZYMES THAT DEGRADE BARLEY GUMS.

IV. Studies of Varietal Differences in Endo-beta-polyglucosidase Activity[1]

E. J. Bass and W. O. S. Meredith

ABSTRACT

Cytolytic activities of the green malts of 9 barley varieties grown at 4 stations in 1954 were determined viscosimetrically. Cytolytic activity was a pronounced varietal characteristic. In general, high activities were associated with satisfactory malt properties, and low activities with unsatisfactory malt properties. The variety Br. 3833, whose cytolytic activity was significantly lower than the standard variety, nevertheless produced a high quality malt. This apparent anomaly must be resolved before the enzymatic assay can be considered as a useful test for prediction or control of quality.

Previous studies (4, 5) have indicated that the primary enzymatic attack on endosperm cell wall materials of barley is due to the action of an endo-beta-polyglycosidase system. The activity of this enzyme system on cellular polysaccharides can be determined viscosimetrically (3, 4).

Interest in cytolytic enzymes, which may degrade cell walls, has been sustained by the possibility that cytolytic activity during germination may influence the degree of modification. Recent European studies of various two-rowed barley varieties (7, 8, 9) have shown that the cytolytic activity of barley bears little relation to malting quality. The apparent failure of barley cytolytic activity to reflect malting quality is attributed by Thunaeus and Sandegren (9, p. 81) to differences in maturation of the barley. These differences are less likely to affect the cytoltyic activity of green malt, which may therefore be more satisfactory than barley for studying varietal differences in cytolytic activity.

This paper presents a study of the cytolytic activities of the green malts of nine six-rowed barley varieties grown at four Canadian stations in 1954. The experiments were designed both to test the possibility that the cytolytic activity of green malt is a varietal characteristic related to malting quality, and to assess the problems involved in a larger-scale varietal study.

Materials and Methods

The nine barley varieties selected for this study have a range of malting quality from good to poor. Montcalm and O.A.C. 21 were in-

[1] Manuscript received October 24, 1955. Paper No. 153 of the Grain Research Laboratory, Board of Grain Commissioners for Canada, Winnipeg, and No. 333 of the Associate Committee on Grain Research (Canada).

cluded as standard varieties of high malting quality; U.M. 570, Sask. 5203, U.M. 1623, and Br. 3833 represented varieties of promising malting quality; Br. 3139, Vantage, and Scott 169 were selected for poor malting quality. Samples of the nine varieties from the 1954 crop were obtained from the experimental station at Morden, Manitoba, and from Melfort, Tisdale, and Saskatoon in Saskatchewan. All samples had satisfactory germination. Five samples germinated 91 to 94%; the remainder germinated 95 to 100%.

To minimize possible malting errors, samples were steeped and germinated in two lots. Each lot contained 20-g. samples of each variety from two stations. The barleys were steeped at 10°C. for 48 hours in perforated plastic containers of approximately 1-pint capacity. After steeping, the samples were germinated[2] in the same containers for 6 days at 12°C. Each sample of green malt was then divided into two equal portions, and stored in a deep freeze at − 18°C. until used.

The analysis of the samples of green malt was arranged to average out variations in cytolytic activity due to enzyme instability during storage of the green malt, and variations due to day-to-day changes in initial substrate viscosity. Three to five subsamples were removed from the freezer daily in a predetermined order, so that the average storage times for all samples were equal. Each sample was then extracted as described below, and extracts were stored in a water bath maintained at 30°C. (±0.2°C.) until used. Cytolytic activities of the extracts were determined in duplicate, with a time interval of about 1 hour between duplicate determinations. This procedure yielded four determinations of cytolytic activity for each barley variety from each station — a total of 144 determinations. The standard analytical error for a single determination was ± 0.035 units, and the standard sampling error for a single determination was ± 0.057 units. These errors can probably be considerably reduced in further studies by slight modifications in the procedure.

Extracts of the green malts were prepared by macerating each portion of green malt with 80 ml. of 0.1 M sodium acetate-acetic acid buffer (pH 4.5) in a Waring Blendor. The components were mixed for two 2-minute periods, with a 5-minute interval between mixings to prevent excessive heating. The mixture was then centrifuged, and the supernatant liquor was filtered through Whatman No. 12 fluted filter paper. A 10-ml. aliquot of filtrate was diluted to 100 ml. with distilled water, and 1 ml. of the final solution was used to determine cytolytic activity.

[2] Barley samples were steeped and germinated at the Barley Improvement Institute, Winnipeg. The malting equipment is described elsewhere (1).

The substrate in this study was barley beta-polyglucoside, described previously (6). Substrate solutions consisted of 1 g. of gum (as is) per 100 ml. of 0.1 *M* sodium chloride solution, with 4 drops of toluene added to inhibit bacteria. Sufficient substrate was prepared for 4 days' analyses, and the solution was stored in a refrigerator at 7°C. when not in use. Substrate viscosity changed by less than 1% during storage for 5 days.

The viscosimetric procedure for determining cytolytic activity was essentially as described previously (4). Enzyme, buffer, and substrate were conditioned to 30°C., then mixed in the proportion of 1 ml. enzyme, 2 ml. buffer, and 7 ml. substrate. Five-milliliter aliquots of each reaction mixture were pipetted into Ostwald Viscosimeters immersed in a 30°C. (± 0.1°C.) water bath, and efflux times were measured at intervals over a period of about 45 minutes. Reaction data were then plotted as reciprocal specific viscosity vs. time. Zero reaction time was taken as the time of addition of substrate to enzyme and buffer. Enzyme activity was arbitrarily defined, as before, as 40 times the slope of the linear reaction curve. This activity was corrected for the moisture content of the original barley.

Results and Discussion

Table I shows the cytolytic activities of the green malts of the 36 barley samples. The raw data were subjected to analysis of variance, and the results are shown in Table II. Varietal differences in cytolytic activity are highly significant. The necessary differences in the means of varietal activities is 0.076 units. Thus, Montcalm and U.M. 570 have cytolytic activities significantly higher than O.A.C. 21 (the standard of

TABLE I

CYTOLYTIC ACTIVITIES OF THE GREEN MALTS OF THE NINE BARLEY VARIETIES

VARIETY	CYTOLYTIC ACTIVITIES[a] OF GREEN MALTS, DRY BASIS				
	Melfort	Morden	Tisdale	Saskatoon	Mean
Montcalm	0.54	0.62	0.55	0.52	0.56
U.M. 570	.57	.66	.53	.35	.53
Sask. 5203	.49	.56	.49	.50	.51
U.M. 1623	.50	.56	.50	.42	.49
O.A.C. 21	.43	.56	.45	.33	.44
Br. 3833	.38	.40	.35	.22	.34
Br. 3139	.29	.39	.30	.16	.28
Vantage	.21	.33	.18	.24	.24
Scott 169	.11	.36	.12	.16	.19
Mean	0.39	0.49	0.39	0.32	0.40

[a] Means of four determinations.

TABLE II

ANALYSIS OF VARIANCE OF CYTOLYTIC ACTIVITY DATA

VARIANCE	DEGREES OF FREEDOM	MEAN SQUARES
Due to:		
Varieties	8	0.3064**
Stations	3	0.1787**
Varieties × stations	24	0.0120**
Analytical error	72	0.0012
Sampling error	36	0.0033

TABLE III

COMPARISON OF CYTOLYTIC ACTIVITIES WITH STANDARD MALT ANALYSES

VARIETY	CYTOLYTIC ACTIVITY	PERCENTAGE BARLEY N	PERCENTAGE MALT EXTRACT	INDEX OF PROTEIN[a] MODIFICATION	SACCHARIFYING ACTIVITY
					°*L*.
Montcalm	0.56	2.28	74.2	38.7	181
U.M. 570	.53	2.42	74.3	35.6	233
Sask. 5203	.51	2.41	73.3	38.8	197
U.M. 1623	.49	2.34	74.6	39.2	185
O.A.C. 21	.45	2.42	73.3	35.6	164
Br. 3833	.34	2.34	75.1	37.6	156
Br. 3139	.28	2.40	68.3	29.9	100
Vantage	.24	2.42	68.2	27.3	101
Scott 169	0.18	2.44	69.8	31.0	154

[a] Index of protein modification $= \frac{\text{wort N} \times \text{extract}}{\text{barley N}}$

malting quality); Sask. 5203 and U.M. 1623 are equal to O.A.C. 21; the remaining varieties have cytolytic activities significantly lower than that of O.A.C. 21. The range of nitrogen contents was too small to determine whether cytolytic activity is solely a varietal characteristic or whether activity is also dependent on nitrogen content.

The mean cytolytic activities of the nine varieties are compared with varietal means of other malt properties[3] in Table III. With the exception of Br. 3833, there is a loose relation between cytolytic activity and the other malt properties. A similar relation was observed between cytolytic activity and ease of malting. Thus, in general, high cytolytic activities were associated with satisfactory malt properties, and low activities were associated with unsatisfactory malt properties.

The variety Br. 3833, which was significantly lower in cytolytic activity than O.A.C. 21, but high in quality as determined by standard malt analysis, appears to cast some doubt on the relation between

[3] These data are reproduced from the Report on Malting Quality of Barley Varieties, 1954, by permission of the Barley Improvement Institute, Winnipeg.

cytolytic activity and malt quality. However, as the activity of this hybrid was approximately 75% of the activity of O.A.C. 21, Br. 3833 may well represent varieties with a borderline level of cytolytic activity that is compensated by some other mechanism.

Van Roey and Hupe (12) also expressed some doubt about the relation between cytolytic activity and malt quality. These investigators reported that a Czech barley, with otherwise satisfactory malt properties, had lower cellulase activity than that of a Polish barley with inferior malt properties. Cellulase activity of the Czech barley was consistently lower than the Polish barley during the germination period, and the Czech green malt was only 25% as active as the Polish green malt. On the basis of these results and of barley cellulose determinations on several European two-rowed barley varieties, Van Roey and Hupe concluded that cytolytic activity is completely unrelated to malt quality. This conclusion contradicts the present findings, hence a possible explanation for the disagreement was sought.

One obvious reason for conflicting results may be the choice of substrate. Modocoll M (11), the artificial substrate used by Van Roey and Hupe in determining cellulase activity, is an ethyl hydroxyethyl cellulose in which the substituting groups may well impede the action of cytolytic enzymes. The natural beta-polyglucoside used by us is an unsubstituted polysaccharide containing both 1:4- and 1:3-beta-glucosidic linkages (2) Consequently if the natural substrate is degraded by a single nonspecific endo-beta-polyglucosidase, this enzyme would not be completely characterized by its action on Modocoll M, which presumably contains only 1:4-beta-glucosidic linkages. Similarly, if the natural substrate is degraded by two specific endo-polyglucosidases, only one of these would attack Modocoll M, and again enzyme activity on Modocoll M would not completely characterize the total endo-beta-polyglucosidase activity. Hence in addition to a possible retardation of cytolytic action by substituting groups on the molecule of Modocoll M, there is the danger that the enzymatic degradation of the artificial substrate may not reflect the natural enzyme-catalyzed reaction.

It appeared worthwhile, therefore, to compare the kinetics of the enzymatic reactions on the two substrates. Substrate solutions of predetermined concentration were prepared to yield reaction mixtures of approximately equal initial viscosity. A 3% solution of Modocoll M in 0.1 *M* sodium acetate-acetic acid buffer (pH 5.0) and a 1% solution of beta-polyglucoside in 0.1 *M* sodium chloride proved satisfactory. An extract of Montcalm green malt was prepared next. Aliquots of this extract were diluted with distilled water to yield solutions containing 2 to 100 ml. of extract per 100 ml. of solution. Reaction mixtures with

Modocoll M as substrate contained 7 ml. of substrate and 3 ml. of enzyme; reaction mixtures with beta-polyglucoside as substrate contained 7 ml. of substrate, 2 ml. of 0.1 *M* sodium acetate-acetic acid buffer (pH 4.5), and 1 ml. of enzyme. Initial specific viscosities of reaction mixtures containing Modocoll M and beta-polyglucoside were 24.5 and 26.5 respectively. Reaction mixtures were at the optimum pH for the respective reactions, pH 5.0 and 4.7 (4, 11). Reaction curves were obtained by plotting reciprocal specific viscosity vs. time, and enzyme activity was defined as 40 times the initial slope of reaction curves.

Reaction curves for both substrates were reasonably similar, but the two substrates differed considerably in sensitivity to green malt enzymes. With Modocoll M as substrate, reaction curves were slightly curvilinear but approximated straight lines initially. Initial slope was directly proportional to enzyme concentration. With beta-polyglucoside as substrate, reaction curves were linear, and their slopes were directly proportional to enzyme concentration. However, enzyme concentrations necessary to produce reaction curves of equal initial slopes were widely different. For example, 3 ml. of green malt extract per 10 ml. of reaction mixture had a viscosity-reducing activity of 0.023 units on Modocoll M. From the activity-concentration curve on beta-polyglucoside, an equivalent activity is obtained when enzyme concentration is 0.006 ml. of extract per 10 ml. of reaction mixture. Thus the ratio of enzyme concentrations necessary to produce the same activity on Modocoll M and beta-polyglucoside solutions is 500:1. Hence there is little doubt that Modocoll M is more resistant than beta-polyglucoside to enzymatic attack. This suggests that there may be a significant difference between the mechanisms of the two enzymatic reactions.

Although there is thus some reason to doubt whether the cellulase determinations of Van Roey and Hupe (12) were a useful measure of cytolytic activity, these experimenters subsequently provided a possible explanation for the anomalous behavior of their Czech barley. Van Roey and Hupe considered the possibility that their cellulase may not be the only enzyme responsible for cytolysis, but they preferred to study, first, the role of the substrate in determining ease of malting. They showed that the Czech barley, with low cellulase activity, yielded less barley gum than the Polish barley with high cellulase activity. They therefore concluded that low gum content could compensate for low cytolytic activity, a possibility suggested earlier by Preece, Aitken, and Potter (10), who have used a barley beta-polyglucoside as substrate. This suggestion merits further investigation as it may well be the reason for the anomalous behavior of Br. 3833.

In summary, then, the endo-beta-polyglucosidase determination on green malt appears to be a pronounced varietal characteristic which, in general, is related to malting quality. Some anomalies, in which relatively low cytolytic activities are associated with otherwise desirable malting properties, are likely to be encountered in plant breeders' material. Further investigation is necessary to determine the cause of anomalous behavior before this enzymatic assay can be considered as a useful test for prediction or control of quality.

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A SMALL-SCALE DOUGH MIXER FOR USE IN WHEAT QUALITY WORK[1]

H. Miller, J. Edgar, and A. G. O. Whiteside[2]

ABSTRACT

The vertical conical mixer described is capable of mixing a 5-g. dough in 1 minute or less. It is based on a light kitchen-type mixer and has a single modified beater arm. The mixing bowl has a 50° center, which lessens the mixing area, intensifies the mixing, and keeps the dough in the path of the rotating spindles. The bowl fits into a temperature-controlled base which prevents chilling of the dough. Characteristics of the dough may be observed during mixing.

Small-scale dough mixers for use in wheat quality work are, as the name implies, apparatus scaled down from larger mixers or in some cases, commercial-size prototypes. If the reduction in size is not too great, it is reasonable to assume that a close correlation in mixing efficiency between the two types will exist.

One of the earlier workers to report on what was then considered to be a small-scale mixer was Jago (1). He used a Werner and Pfleiderer small doughing machine for his routine baking tests. A sample of flour weighing 560 g., i.e., 2 g. for every pound in an English sack of flour, was thoroughly mixed into a dough by means of this apparatus. Although the horizontal experimental mixer has been largely superseded by the vertical spindle or pin type mixer, an excellent example of its efficiency is still to be seen in the Brabender Farinograph.

The vertical pin type mixer developed by Swanson and Working (5) was an improvement on the earlier Bachmann mixer. Small-scale refinements of this principle are to be seen in the mixograph and in the equipment described by Shogren and Shellenberger (4). Another modification of the spindle mixer was developed by Malloch (2), who introduced a stem in the mixing head to reduce the mixing area and curved spindles to keep the dough near the bottom of the cup.

In constructing a mixer that will mix 5-g. doughs for the expansion test described by the authors (3) or for any other small-scale test, a number of factors must be kept in mind. Such a mixer has to be simple in design and in operation. It is desirable that the mixing operation be under observation at all times to permit stoppage if overmixing

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[2] Canada Department of Agriculture.

ensues. The mixer must be efficient enough to mix a dough thoroughly and quickly. The dough must be readily removed from the mixer. These points were taken into account in constructing the mixer described in this paper.

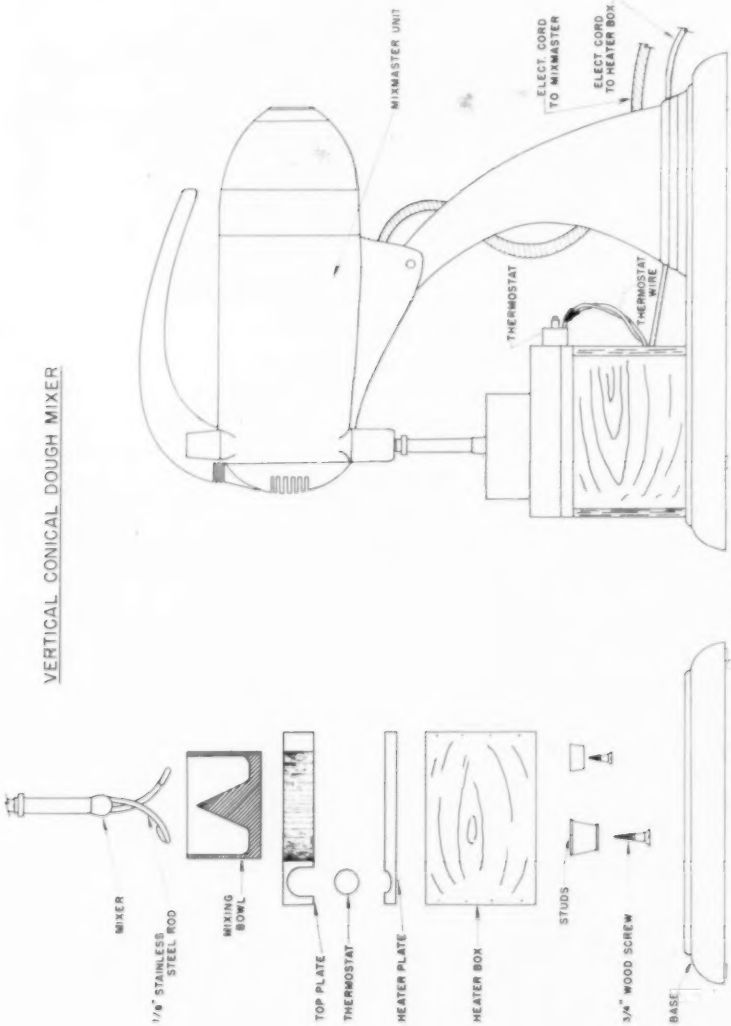

Fig. 1. Diagram showing the component parts of the small-scale dough mixer.

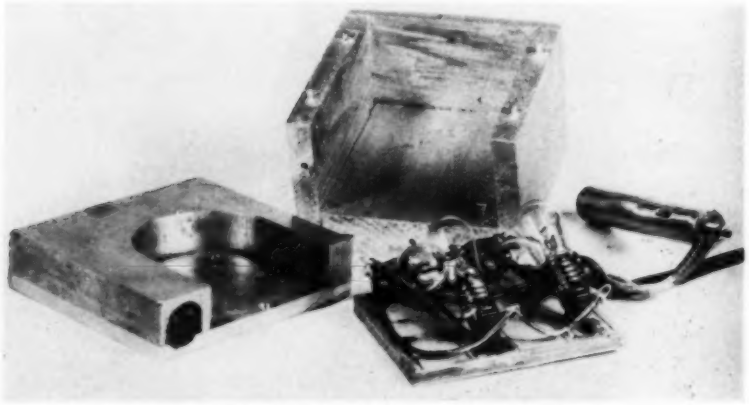

Fig. 2. Holder for mixing bowl and heater box assembly.

Description of Small-Scale Dough Mixer

In the diagram, Fig. 1, the construction of the component parts of the dough mixer is shown. A household-type mixer is used as the power source. The mixing arm is constructed by removing the beater blades from the cake mixer beater and substituting two stainless steel spindles. These are fabricated from 1/8-inch rods and curved in descending counterclockwise spirals following the outline of the center cone in the mixing bowl. The mixing bowl is turned from brass bar stock and is tinned to prevent metallic contamination of the dough. The 50° cone in the bottom of the bowl forms an endless troughlike mixing area when the dough is mixed.

The mixing bowl sits on a holder consisting of two parts: the upper part, an aluminum casting with a recessed groove to hold the bowl; the lower part, a piece of 1/4-inch aluminum plate. Together they form the top of the heater box which is held in place on the box by four pegs and a single wood screw. Warming of the aluminum plate and the mixing bowl is achieved by the use of four 6-watt pilot lamps hooked up in series and connected to a thermostat recessed into the holder. The sockets of the lamps are mounted on a piece of plywood. The back of the heater box is easily removed to allow periodic examination of the lamp assembly, which slides out without removing the metal top. The heating box was found necessary when it was noted that narrow temperature differences influenced the character of a small dough. Figure 2 shows the bowl holder and heater box assembly.

Operation of the Mixer

Figure 3 illustrates the assembled mixer and the mixing operations

under way. The head is raised when the flour is placed in the mixer and lowered into position by moving the bowl out slightly. The solutions are added from micro pipettes and the mixing is commenced. The mixer is run at its lowest speed, which turns the mixing arm at approximately 380 r.p.m. The sides of the bowl are cleaned by use of a plastic spatula while the bowl is rotated slightly by hand. After about 20 seconds of mixing the dough forms into a ball and development continues from this point forward. A strong dough will be well mixed in 60 seconds, but a weak dough may require only 30 seconds. The dough ball is readily removed from the bowl at the end of the mixing and is rounded in the hand.

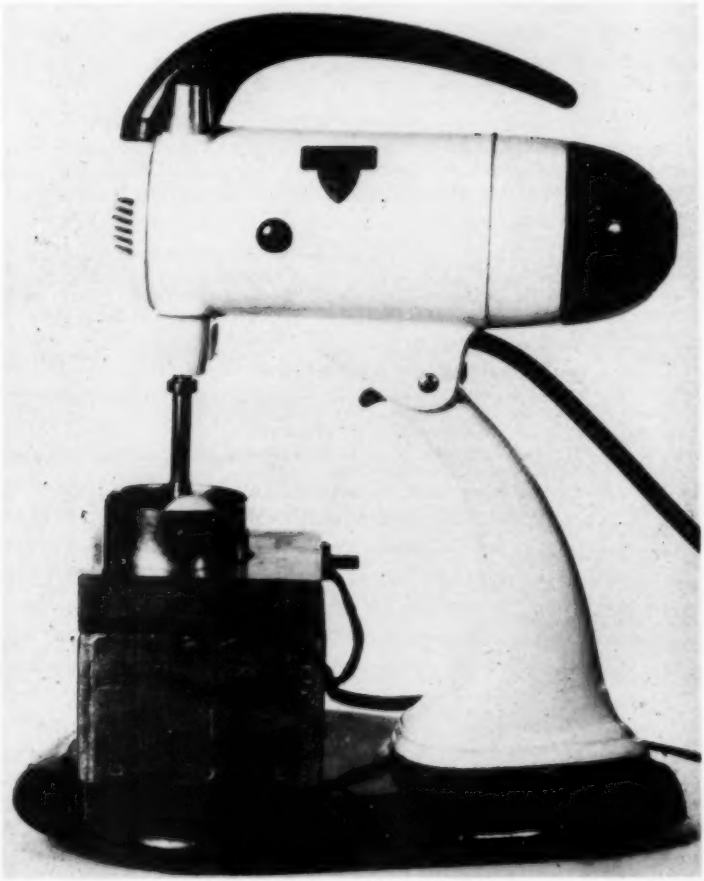

Fig. 3. The assembled small-scale dough mixer.

A 2-minute mixing schedule is currently used in mixing 5-g. flour doughs. Using three micro pipettes, the ingredients, in the form of a salt-sugar-improver solution, a yeast suspension, and water, are added to the flour in the mixing bowl. For a given series of flours a standard absorption is used and any flour which deviates in this absorption is recorded and scored accordingly, or an adjustment may be made on a second test of that flour. In the mixing of flours, quality differences will be revealed even though the sample is small.

The mixer described in this paper has been found suitable in the mixing of many 5-g. flour doughs in the authors' laboratory. It is a modified version of the mixer used in conjunction with the expansion test for the experimental work described by Miller *et al.* (3), and its efficiency may be assessed by the low replicability error reported for the expansion test.

Acknowledgments

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A SIMPLE METHOD FOR DETECTING CONTAMINATION OF WHEAT BY RODENT URINE[1]

J. W. Laakso, Madelyn Ferrigan, M. O. Schultze, and W. F. Geddes

ABSTRACT

Contamination of wheat by rodent urine can be readily detected by treating a large number of individual kernels on a spot plate made from clear plastic with buffered urease solution (pH 6.8) for 5 minutes, followed by addition of Nessler's reagent which forms a yellow to orange-colored complex with the ammonia which is liberated from the urea present. Acuity of differentiation between normal and contaminated kernels is increased by examination with an ultraviolet lamp or by viewing the color with incandescent light which has passed through a blue or blue-green filter. Wheat kernels dipped into a 0.1% solution of urea or into rat urine diluted with 50 parts of water, after draining and drying, gave positive tests.

The accuracy of the method has been demonstrated by tests on samples containing known percentages of contaminated kernels. Adult rats allowed to roam over the surface of wheat contaminated approximately 10,000 kernels per rat per day when water was supplied.

Urine is readily detected on grain and flour sacks by its fluorescent properties upon exposure to ultraviolet radiation. However, tests in the Minneapolis laboratories of the U.S. Food and Drug Administration[2] have shown that the usual blue-white fluorescence is not clearly visible on wheat contaminated with urine, and no chemical treatment of the wheat was found which would satisfactorily enhance the fluorescence. Urea, one of the main solid constituents of urine, yields ammonia and carbon dioxide upon treatment with urease solution, and this reaction is utilized in two methods for the detection of urea on foods and food containers described in the 8th edition of *Official Methods of Analysis of the Association of Official Agricultural Chemists* (1). In one method the ammonia is detected by means of a hanging drop of platinic chloride solution; in the other, a test paper is employed which contains urease and the acid-base indicator, thymol blue. These methods are not convenient for determining the percentage of kernels contaminated with urine in a parcel of grain, because of the large number of individual kernels which must be examined to provide a reliable sample.

The method described in this paper is based on the detection of ammonia liberated from individual kernels by urease solution through its reaction with Nessler's reagent to yield a yellow to orange-colored

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[2] Private communication from George E. Keppel.

complex. The sensitivity and accuracy of the test were investigated. The results of its application to five commercial wheats containing fecal pellets are presented; in addition, the method was employed in determining the extent of urinary contamination of wheat when rats were allowed to roam over the surface of the grain.

Method for Detection of Urea on Wheat Kernels

Apparatus and Reagents: (1) Test plate. Into a sheet of clear plastic 15 x 11½ x ¼ in., drill 712 depressions each 3/16 in. deep and 1/4 in. in diameter. (2) Nessler's reagent: Prepare the modified Nessler-Folin reagent (2,3) as follows: Add 20.15 g. of mercuric iodide to 10 ml. water containing 15 g. of potassium iodide and warm, if desired, with shaking to obtain rapid solution. Cool and test the solution by adding a few drops to 1 ml. of a 1% solution of soluble starch. Unless the starch test for iodine is obtained, the solution may contain mercurous compounds; if necessary, to the remaining solution add a few drops of an iodine solution (prepared by dissolving 11.25 g. of iodine in 10 ml. of water containing 15 g. of potassium iodide) until a faint excess of free iodine can be detected with the starch test. Dilute to 100 ml. and mix well. To 487.5 ml. of an accurately prepared 10% sodium hydroxide solution now add the entire solution of mercuric iodide prepared above. Mix thoroughly and allow to clear by standing. (3) Urease solution. Suspend 2.0 g. of jack bean urease preparation (Sigma Chemical Co., St. Louis, Mo.) in 100 ml. of a buffer solution (pH 6.8) containing 2.4 g. of dibasic potassium phosphate and 1.4 g. of monobasic potassium phosphate. Prepare the urease solution fresh daily; older solutions yield detectable amounts of color when treated with Nessler's reagent.

Procedure. Into each of 702 depressions in the test plate, place one kernel of wheat from the sample under test. In each of 10 depressions in the center of the plate place a kernel of wheat from a sample known to be free of urinary contamination. To each kernel add one drop of the 2.0% buffered urease solution from a pipette. After 5 minutes add one drop of Nessler's reagent and observe whether a yellow to orange color develops. Detection of the color is facilitated by examining the treated kernels with an ultraviolet lamp or with incandescent light which has been passed through a light filter of blue or blue-green plastic paper placed underneath the test plate. Wheat which has not been contaminated with urine does not show any appreciable color and the kernels in the center of the spot plate can be used for purposes of comparison.

Sensitivity. The high sensitivity of this qualitative procedure is

indicated by the fact that a positive test was given by wheat kernels which had been dipped into a solution containing 1 part of urea per 1000 parts of water, then removed and permitted to drain and dry. Likewise, a positive color test was obtained with wheat kernels which had been dipped in rat urine diluted fiftyfold with distilled water and then allowed to drain and dry.

Accuracy. As a test of the accuracy of the method, three mixtures were prepared by incorporating into a known number of presumably uncontaminated kernels a known number of kernels which had been dipped into rat urine, then drained and dried. The mixtures were so prepared as to provide levels of contamination of 0.5, 2.0, and 8.0% by count. Ten subsamples of each thoroughly mixed sample were then tested, with the results shown in Table I. The mean numbers of contaminated kernels found are in excellent agreement with the numbers which are present.

TABLE I

DETECTION OF URINE IN WHEAT CONTAINING A KNOWN PROPORTION OF CONTAMINATED KERNELS BY THE UREASE-NESSLER'S REAGENT PROCEDURE

LEVEL OF CONTAMINATION	MEAN NUMBER OF CONTAMINATED KERNELS PER 697[a]		STANDARD ERROR IN PERCENT OF MEAN
	Calculated	Found	
%			%
0.5	3.5	3.7±0.58	15.7
2.0	13.9	13.3±0.78	5.9
8.0	55.8	57.0±2.96	5.2

[a] Mean contaminated kernels found are based on ten determinations.

Applications of the Color Test for Urea

Counts on Commercial Wheats. The results of replicate counts on samples of commercial wheats which contained rodent fecal pellets and were supplied by the Minneapolis Grain Exchange are summarized in Table II. Although there was no relation between the number of fecal pellets and the number of wheat kernels found to be contaminated with urine, urine was present in all of the samples contaminated with rodent pellets.

Extent of Urinary Contamination of Wheat by Rats Roaming on the Surface. To determine the extent of contamination of wheat by rodent urine, two rats (each weighing 250 g.) were permitted to roam for 48 hours in a drum (surface area 2.46 sq. ft.) containing a layer of 38.6 kg. of wheat kernels. Distilled water was supplied. This wheat was then thoroughly mixed and 702 kernels were tested for the presence of urea by the urease-Nessler's reagent procedure. From 11 sub-

TABLE II

DETECTION OF URINE IN COMMERCIAL WHEATS CONTAINING DIFFERENT NUMBERS OF FECAL PELLETS

NUMBER OF PELLETS PER 1000 G.	NUMBER OF COUNTS	MEAN NUMBER OF CONTAMINATED KERNELS[a]	STANDARD ERROR, IN PERCENT OF MEAN
			%
3	21	7.0±0.76	10.8
3	21	14.7±1.13	7.7
5	10	2.9±1.02	35.1
6	17	6.9±0.60	8.7
11	20	0.6±0.42	7.0

[a] 697 kernels were examined in each sample.

samples of 702 kernels each, a mean count of 25.4 contaminated kernels was obtained with a standard error of ±2.7. This indicates that 3.62% of the kernels were sufficiently contaminated to permit detection by this procedure. Assuming an average kernel weight of 35 g. per 1000, this percentage is equivalent to about 40,000 kernels contaminated by two rats in 48 hours or about 10,000 kernels per rat per day. This is a surprisingly large figure in view of the fact that the urinary excretion of rats of this weight was found to be approximately 8.0 ml. per day when fed on a diet consisting of 82 parts wheat, 17 parts dried whole milk powder, and 1 part sodium chloride with water supplied. However, measurements of the surface tension of rat urine showed it to be about one-half that of distilled water; as a result, urine penetrates the interstices between the grains and spreads readily from kernel to kernel. The spreading power of rat urine can be demonstrated through subcutaneous implantation of pellets of indigo carmine (100 mg.) to impart a blue color to the urine. The surface tension or volume of the urine was not altered by this treatment. Experiments showed that the urine from one rat roaming on the surface of wheat (2.46 sq. ft.) for 30 to 48 hours would penetrate the grain to a depth of at least 3.1 in. Counts of kernels with blue stains showed that rats weighing from 290 to 300 g. contaminated 4560 to 5350 kernels of wheat with urine per 24 hours. These values are approximately one-half that obtained by the urease-Nessler's reagent procedure. While the urinary output of rats is subject to considerable fluctuation, the difference between the results indicates that the method described here is the more sensitive.

Discussion

The urease-Nessler's reagent procedure is applicable to the examination of grain contaminated with urine under natural conditions.

Under laboratory conditions, wheat contaminated with urine gave a strong positive test after nine months' storage; it therefore does not appear that urea will decompose sufficiently to escape detection by this procedure when stored at moisture levels which do not permit spoilage of the grain to occur.

Acknowledgment

This research was supported in part by a grant from the Minneapolis Grain Exchange, Minneapolis, Minnesota.

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PYRETHRINS-PIPERONYL BUTOXIDE AS A RESIDUAL TREATMENT AGAINST INSECTS IN ELEVATOR BOOTS[1]

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ABSTRACT

In a flour mill, elevator boot stocks treated with 2.5% pyrethrins and 25% piperonyl butoxide at 1 ml. of insecticide per 50 g. of stock had fewer live adults and pupae of the confused flour beetle, *Tribolium confusum* Duval, and the flat grain beetle, *Laemophloeus pusillus* (Schönh.), than untreated boot stocks 5 weeks after treatment. Treated stock induced paralysis in *T. confusum* and reduced movement to other milling equipment. Flour passed over a treated boot showed a faint odor of the insecticide in loaves baked from the first sample, but the slight odor on subsequent samples was not noticeable after fermentation and baking.

Elevator boots in flour mills often harbor insects in "dead" stock. These insects may be carried to other equipment in the mill by cereal stocks passing through infested boots. Control measures are directed at killing the insects in the boots and other equipment by local fumi-

[1] Manuscript received August 23, 1955. Contribution No. 3345, Entomology Division, Science Service, Department of Agriculture, Ottawa, Canada.
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gation. However, most fumigants evaporate rapidly and treated sites are then subject to reinfestation (4).

A residual contact insecticide applied to elevator boot stocks may be expected to give longer-lasting protection against insects than most fumigants. Pyrethrins-piperonyl butoxide was considered to be an appropriate insecticide for this purpose. It has been used for the protection of stored grain against insects (2, 5) and has low mammalian toxicity (3).This paper describes experiments carried out in a flour mill to test the effectiveness of pyrethrins-piperonyl butoxide as a residual treatment in elevator boots.

Materials and Methods

Three aspects of the experimental treatments were studied: first, comparison of different concentrations of pyrethrins-piperonyl butoxide applied in elevator boot stocks; second, effect of the treatment on the movement of insects in an experimental elevator; third, effect of the treatment on baking quality of flour.

Different Concentrations. Comparative effectiveness of three treatments was assessed with 24 elevators selected at random in a flour mill. Three groups of six elevator boots were treated with different concentrations of pyrethrins-piperonyl butoxide. One group of boots remained untreated. The cereal stock was cleared from each boot and sampled with a standard-volume container. All samples taken before the treatments, and 5 weeks later, were incubated at 75°F. and 75% R. H. for 2 weeks and then examined for live adults and pupae of the confused flour beetle, *Tribolium confusum* Duval, and the flat grain beetle, *Laemophloeus pusillus* (Schönh.). These two species made up 99% of the boot population sampled. The only other species found in the boots was the cadelle, *Tenebroides mauritanicus* L.

The pyrethrins-piperonyl butoxide concentrate was diluted with deodorized kerosene to give the following solutions (w:v): 2.5% pyrethrins and 25% piperonyl butoxide; 1.5% pyrethrins and 15% piperonyl butoxide; 0.5% pyrethrins and 5% piperonyl butoxide. The application rate was 1 ml. per 50 g. of stock. Each solution was applied to the stock of six elevator boots.

Usually between 6 and 8 lb. of stock from each boot were treated; this was the quantity that would not overload most boots when the stock was returned. The stock was spread on the floor in a layer 2 in. deep, sprayed with a measured volume of insecticide, mixed thoroughly, and returned to the boot.

Movement of Insects. Experiments carried out at the Winnipeg

laboratory had shown that adults of *T. confusum* become paralyzed and are unable to mate or oviposit when placed on flour impregnated with pyrethrins-piperonyl butoxide at certain dosages. Studies were, therefore, made to compare the behavior of insects in an elevator boot that contained untreated flour with that in one with flour treated with pyrethrins-piperonyl butoxide. An elevator in a flour mill was modified to form a closed system in which flour, poured in the spout and elevated to the top, could be either removed or recirculated through the boot and elevator.

The elevator boot was charged at the beginning of each test run with untreated flour, or flour treated with 2.5% pyrethrins and 25% piperonyl butoxide (w:v) at 1 ml. per 50 g. of flour. Two hundred adults of *T. confusum* were distributed throughout the boot stock. The beetles were marked with a spot of colored enamel on the left elytron for identification. The infested stock was left undisturbed in the boot for 5 minutes or 3, 12, or 24 hours before the elevator was started. The elevator was allowed to run for 1 hour. Stock moving through the spout was sampled at intervals and sieved to remove insects. After each run the free stock in the elevator and spouts and the "dead" stock in the boot were removed and sieved for insects.

Baking Quality. Tests were also made to find out whether the baking quality of flour that had passed through a treated boot was adversely affected. Six pounds of flour were treated with an oil solution containing 2.5% pyrethrins and 25% piperonyl butoxide at 1 ml. per 50 g. of flour. The treated flour was placed in the experimental boot previously described. Fresh flour was placed in the elevator and samples of the moving stock were taken at the beginning of the run and at 2-minute intervals for 10 minutes. The samples were stored in paper bags that were kept closed before baking. The flour was scored for odor, color, baking quality, and loaf volume.

The general method given in *Cereal Laboratory Methods* (1) was used for baking the bread, but, since loaves baked from 100 g. of flour would be too small for the organoleptic tests, the method was scaled up to 330 g. of flour. Furthermore, to compensate for the addition of 10 p.p.m. potassium bromate and 0.3 g. of chlorine dioxide per bbl., the amount of yeast was cut from 3 to 2%.

Analysis of Data. Analysis of covariance was carried out on data collected to compare treatments. An appropriate transformation, log $(x + 1)$, was applied to the data before analysis. The mean numbers of insects given in Table I were obtained by taking the antilog of $\bar{y} + 1.15\ s^2y$, where $\bar{y}$ is the mean of the logarithms and s^2y is their error mean square.

Results and Discussion

Table I gives the mean numbers of live insects obtained from the boot stocks before treatment and 5 weeks after treatment. Analysis of covariance indicated significant differences between the treatments and the check.

Student's *t* test of data 5 weeks after treatment showed a highly significant difference between untreated boots and those treated with 2.5% pyrethrins-25% piperonyl butoxide ($P<0.01$), and a significant difference between untreated boots and those treated with 1.5% pyrethrins-15% piperonyl butoxide ($P<0.05$). There was no significant difference between these two treatments ($P=0.4$), or between untreated boots and those treated with 0.5% pyrethrins-5% piperonyl butoxide ($P=0.27$). The difference between boots treated with 2.5% pyrethrins-25% piperonyl butoxide and with 0.5% pyrethrins-5% piperonyl butoxide was significant at the 9% level.

Table I shows that there were fewer insects in the untreated boots 5 weeks after than before treatment. A possible explanation is that, when the boots were first cleaned, less stock was replaced than was removed, to prevent blockage of conveyor cups. Hence, the numbers of insects in boots under treatment would be similarly reduced. Student's *t* test showed that the reduction of insects in boots treated with 2.5% pyrethrins-25% piperonyl butoxide 5 weeks after treatment was highly significant ($P<0.01$). The reduction of insects in boots treated with 1.5% pyrethrins-15% piperonyl butoxide was significant at the 7% level after 5 weeks. There was no significant reduction of insects in boots treated with 0.5% pyrethrins-5% piperonyl butoxide ($P=0.15$), or in untreated boots ($P=0.5$).

Table II shows that more insects remained in the boot when it

TABLE I

Insects Obtained from Comparable Samples of Elevator Boot Stocks[a]

Treatment[b]	Number of Boots Sampled	Mean Numbers of Insects Obtained	
		Before Treatment	After 5 Weeks
2.5% pyrethrins-25% piperonyl butoxide	6	166.3	20.2
1.5% pyrethrins-15% piperonyl butoxide	6	36.2	15.7
0.5% pyrethrins-5% piperonyl butoxide	6	76.7	39.1
None	6	128.8	96.2

[a] Converted from transformed data.
[b] Applied at the rate of 1 ml. of insecticide per 50 g. of stock.

contained treated flour than when it contained untreated flour. All insects in treated flour showed paralysis and were unable to walk; locomotion was restricted to undulating movements in the flour. Movement of insects into other milling equipment was, therefore, restricted with treated boot stock. This contrasted sharply with the activity of

TABLE II

NUMBERS OF ADULTS OF THE CONFUSED FLOUR BEETLE, *Tribolium confusum* DUV., RECOVERED FROM MOVING STOCK AND FROM STATIC STOCK IN AN ELEVATOR

REST PERIOD BEFORE STARTING OF ELEVATOR	INSECTS RECOVERED FROM MOVING STOCK DURING FIRST HOUR	INSECTS RECOVERED AFTER 1 HOUR		INSECTS NOT RECOVERED	TOTAL
		Boot	Leg		
	Boot Stock Untreated				
5 minutes	53	58	5	84	200
3 hours	43	129	0	28	200
12 hours	25	116	0	59	200
	Boot Stock Treated				
3 hours	3	190[a]	0	7	200
24 hours	4	202	0	0	206

[a] Elevator run 4 days.

insects in the boot when it contained untreated flour. Then, the test insects moved about rapidly when introduced; many were observed climbing up into the elevator legs. Others moved into positions where they were scooped up by the cups and transferred to stock passing through the boot. In practice, these insects would be carried to other equipment in the mill, whereas almost all insects in boot stock treated with pyrethrins-piperonyl butoxide would remain there.

The chi-square test indicated that in untreated flour the numbers of insects recovered from the boot, and from flour in the elevator cups and the spout, was independent of the time the elevator was left idle before being started.

Baking tests of samples of flour passed through treated boot stock showed that a faint odor of the insecticide was noticeable in loaves from the first sample, taken immediately after the elevator was started. A slight odor was detected in the remaining five samples, taken at 2-minute intervals, but this disappeared after fermentation and baking. Loaf volume of samples baked from flour that had been circulated in the elevator for 2 minutes or longer was greater than that for the check samples. This was thought to be due to aeration of the flour while it

was being circulated in the elevator. The insecticide had no specific effect on color or texture of the bread.

The odor imparted to the flour in treated boots is apparently related to the quantity of flour that passes through the boot. Odor, therefore, is not likely to become a problem in treated boots that handle normal quantities of stock, or in boots that carry stocks less susceptible to taint than flour.

In the United States, the Food and Drug Act prohibits the contamination of flour by an insecticide. The establishment of insecticide tolerances, under the Miller Bill, for the treatment of raw agricultural products such as grain is presently being considered. Treatment of elevator boot stocks with any insecticide that might be transferred to the finished product must conform to tolerances that may be established by new legislation.

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The author wishes to thank Mr. H. Bergsteinsson, Chemist, Lake of the Wood Milling Co., Keewatin, Ontario, for conducting the baking tests. Mr. W. Ives, Forest Biology Laboratory, Winnipeg, gave helpful advice on statistical methods. Mr. G. A. Cox, Entomology Laboratory, Winnipeg, assisted in the insecticide applications and insect counts.

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BOOK REVIEWS

Industrial Fermentations, Vol. I, ed. by Leland A. Underkofler and Richard J. Hickey, 565 pp. Chemical Publishing Co., Inc., New York, 1954. Price, $12.00.

The first contact of the reader with this book is through the list of contributors and the commercial concerns or universities where they work. This list precedes the preface, and such prominence is deserved, for on the list are names well known in industrial and academic circles. That the authors have technical information which readers desire can hardly be doubted, and the solid but readable information packed into the 17 chapters comprising the book discloses the good degree of freedom which most of the authors have felt in the presentation of important details.

The fermentations described in Volume I are those in which the product constitutes at least a few percent of the fermented liquid, as compared to the fine fermentations which constitute most of Volume II, namely antibiotic, enzyme, and vitamin production. Other discourses included in Volume II cover ketogenic fermentations, the production of 2,3-butanediol, waste treatments, and maintenance and variation of cultures.

Ten chapters in Volume I are concerned with yeasts. The first deals with the fermentation of grain, principally for the production of alcoholic beverages. The author, whose extensive experience has been in both the biological and the engineering fields, has given a lucid and informative description of the more common procedures, illustrated with flow-sheets and photographs of equipment used at two leading distilleries. Because the author has long been associated with fermentation companies, the discourse on cultures, inoculum, and the main fermentation is adequate but not out of proportion to other important phases, such as milling, cooking of grain mashes, malting and other means of converting starch to sugar, back slopping, contamination, batch and continuous fermentation, distillation, recovery of grain residues, and analytical methods. Chapter 3 deals with procedures concerned with the production of industrial alcohol from molasses, and competitive processes that threaten the industry.

Certain European countries have practiced rather extensively the production of alcohol as well as food and feed yeast from sulfite liquor, which is a waste product in the manufacture of paper. Rather recently, similar plants have been put into operation in the United States and Canada, and there is good possibility that the production of feed yeast from this cheap and abundant source will increase. Processes used for the production of alcohol in specific European and North American plants are presented in chapter 4. It may be noted that the decimal points in the figures given in table 18 for concentrations of fermentable sugars are shifted one space to the left of where they should be. Chapter 5 deals with the production of wood hydrolysates. The hydrolytic procedures are sufficiently strong to convert not only the hemicelluloses, which are the sugar-yielding compounds concerned in sulfite liquor production, but cellulose as well, to yield a more concentrated sugar solution. Considerable experimental work has been done, pilot and full-scale plants have been erected in the United States, and considerable technical know-how has been accumulated. However, the economics involved at the present time are not favorable for production of alcohol from wood hydrolysate. Chapter 10 deals with the production of food and feed yeast, particularly from sulfite liquor. This industry is currently expanding, especially in Wisconsin. The principal yeast involved is *Torulopsis utilis*, a rugged species with tolerance for toxic substances which most other microorganisms cannot stand, and a synthetic ability that supplements media too deficient to permit the growth of the varied microbial flora which gains access to the open Waldhof or similar propagators commonly employed in the process. Thus, the propagators can be operated continuously for months or years without serious contamination troubles. Chapter 9 is concerned with the production of baker's yeast. Certain vital details of the production of active dry yeast have not been disclosed here.

A large amount of information is contained in the chapter on wines, and an adequate treatment of the processes used for vinegar production is recorded in an-

other chapter. Beer is more sketchily treated from the technical side. For example, the reader is not informed on such important points as range of time and temperatures used in the aging of beer.

Five chapters deal with potentially available fermentations which are not used by industry or are being used to a slight extent, because of competition by chemical synthesis from petroleum fractions, because of insufficient uses for the product, or because cheap methods have not been devised for separation of the product from the fermented liquor. These chapters present experimental results and experience with pilot-plant or industrial-scale production where such have been obtained. This group includes the chapters on glycerol, for which the demand is reported as good but a purely chemical process is ready and able to deliver; gluconic and lactic acids which are being produced biologically to supply needs principally in the detergent and food fields, respectively; and fumaric and itaconic acids which can be produced in good yields by molds. Citric acid is a mold fermentation which has made good, and recently done even better, owing to partial conversion from pan to submerged fermentation procedure.

The reviewer remembers the chapter on the butanol-acetone fermentation for the strange "binomials" (*Clostridium saccharo-butyl-acetonicum-liquefaciens-delta*, if you please) and the good, detailed way in which the various modifications of the process are presented. The author of the chapter is not responsible, of course, for the names; they were invented for consumption by patent offices.

Volume I of *Industrial Fermentations* is an excellent compendium of basic knowledge of the 16 fermentations which it describes.

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Industrial Fermentations, Vol. II, ed. by Leland A. Underkofler and Richard J. Hickey, 578 pp. Chemical Publishing Co., Inc., New York, 1954. Price, $12.00.

This volume contains fourteen chapters which, together with the seventeen in Volume I, deal with the most important industrial fermentations and subjects related thereto. The editors, who contributed some of the chapters of this volume, and the other writers are recognized authorities in their respective fields; thus one finds recorded, and well documented with references, a wealth of pertinent and useful information. Students and investigators outside of industrial practices will generally find, in those chapters discussing specific fermentations, information about the microorganisms, including their nutrient and environmental requirements; methods of analysis for the main product formed; the course of the fermentation; and laboratory and large-scale equipment. The first chapter in this category, by L. B. Lockwood, entitled "Ketogenic fermentation processes," deals principally with dehydrogenation or oxidation reactions for the conversion of polyhydric alcohols, sugars, or polyhydroxy acids to other products. The conversion of sorbitol to L-sorbose, an intermediate compound in the commercial manufacture of ascorbic acid, is a main example.

The chapter "Fermentation production of 2,3-butanediol," by G. A. Ledingham and A. C. Neish, is the most comprehensive treatment available of the developmental work connected with this fermentation. Although not in commercial use, the importance attached at one time to this fermentation for the contemplated production of precursors to the manufacture of 1,3-butadiene is attested to by about 500 publications, of which 282 are cited by these authors. The discussion of the probable mechanism for the synthesis of 2,3-butanediol by the reduction of acetoin, and the list of compounds formed from 2,3-butanediol, is interesting.

The production and uses of fungal amylolytic enzymes are thoroughly discussed by L. A. Underkofler, who is recognized as a leading authority in this field. This author has generously supplied detailed information obtained by personal observations of a pilot plant and a semicommercial plant producing fungal amylase by submerged culturing of molds. Microbial enzymes other than fungal amylases are ably reviewed by J. C. Hoogerheide.

Biological processes for the industrial production of riboflavin, particularly with *Eremothecium ashbyii*, *Ashbya gossypii*, and *Clostridium acetobutylicum*, are clearly

described by R. J. Hickey, who contributed much to our knowledge of the first process. The production of other vitamins as primary or byproducts of fermentation processes and the role of microorganisms in the discovery of vitamins have been well described by J. M. Van Lanen.

A vast amount of information on the many facets of the antibiotics industry has been clearly and thoroughly presented in three chapters. They are "The penicillin fermentation" by J. C. Sylvester and R. D. Coghill; "Streptomycin" by H. B. Woodruff and L. E. McDaniel, and "The broad spectrum, polypeptide and other antibiotics" by William H. Peterson and Mary E. Peterson. All of these writers have emphasized the production, isolation, and chemical nature of commercial antibiotics, and the latter writers included a table summarizing the main facts concerning 78 other well-defined antibiotics.

Dextran as a blood-plasma expander recently stimulated extensive research and new information has been given about its production and structure. Also, the synthesis of L-ephedrine and the action of microorganisms on steroids are explained in an interesting chapter by A. R. Stanley and R. J. Hickey entitled "Miscellaneous fermentations."

Industrial food fermentations are typified in an excellent chapter by R. H. Vaughn, who described lactic acid fermentation of cucumbers, sauerkraut, and olives.

The selection of suitable cultures of microorganisms and methods for preserving cultures to retain their desired characteristics for industrial fermentation has been authoritatively described by E. F. McCoy. The paraffin-oil, soil-stock, and lyophilization methods are those in widest use.

Cultural variation and genetics of microorganisms have been discussed by C. C. Lindegren. Industrial microbiologists will probably be most interested in the section dealing with heterokaryosis. The value of this chapter would be greater if it described the presently used physical and chemical technics for inducing cultural mutations.

The book is concluded with an interesting chapter by A. M. Buswell, entitled "Fermentations in waste treatments." Emphasis is placed on the production and uses of methane gas. The sale of about 100,000 tons annually of activated sludge as fertilizer is recorded.

During the time the manuscripts for the chapters of this book were being written, the name of the frequently mentioned Northern Regional Research Laboratory of the U. S. Department of Agriculture was changed to Northern Utilization Research Branch. Through no fault of the writers, both names appear in the book. This change of name does not, however, affect the letters used to designate microorganisms in its Culture Collection. Cultures continue to be identified as, for example, *Penicillium chrysogenum* NRRL 1951.

Volume I includes chapters on alcohol and acid fermentations and the manufacture of yeast.

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Radioisotopes in Biology and Agriculture, Principles and Practice, by C. L. Comar, 1st ed., xiii + 481 pp. McGraw-Hill Book Co., Inc., New York, 1955. Price, $9.00.

Radioactive tracers have unquestionably become one of the most important analytical tools now at the disposal of the experimental biologist. The rapid development of the technics involved in the use of radioisotopes has created a serious need for a critical compilation of information for planning and executing tracer studies. This book has been written in an attempt to fulfill this need, with particular emphasis on the practical application of current knowledge to biological systems.

The basic principles of radioactivity are treated in chapter I in a simple, straightforward manner not likely to discourage the uninitiated, as is so often true in texts dealing with the more detailed aspects of nuclear physics. Numerous examples from such diverse fields as physiology, nutrition, entomology, and soils serve to illustrate how the use of radioisotopes may fit into investigations dealing with biological phenomena.

Lest the reader be lulled into believing that tracer technics may provide an

unequivocal answer to his particular problem, chapter 2 points out the difficulties and pitfalls inherent in tracer studies and how they may be avoided or interpreted. Chapter 3, which is concerned with the practical problem of health physics and radiation protection, does much to allay any fears that the novice may have in undertaking work with radioactive materials, and at the same time will make the reader more appreciative of the importance of a few simple precautionary measures.

Perhaps the most valuable portion of this book will be found in chapters 4 and 5 which describe the facilities and procedures for tracer studies in plants and in laboratory and domestic animals. The author freely admits that no attempt has been made to include all the procedures and technics described in the literature for fear of confusing the uninitiated who would be forced to choose among them. Only those methods which extensive usage has shown to produce satisfactory results have been presented, in sufficient detail to make it unnecessary for the investigator to comb through myriad individual papers for the experimental details he might need.

Chapter 6 summarizes the properties of each radioisotope and describes specific methods for their handling and assay. Chapters 7 to 10 deal with technics which have proved of value in conjunction with radioactive work such as autoradiography, paper chromatography, ion exchangers, and radioactivation analysis.

The reader who wishes to consult the original literature for additional information on specific methods will appreciate the comprehensive list of references cited in the text as well as the list of selected, general references which accompanies each chapter. Valuable adjuncts to this book are the glossary of terms most frequently used in nuclear science, a list of available U.S. Atomic Energy Commission Reports, and a descriptive list of the radioisotopes which can be purchased from the Oak Ridge National Laboratory.

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Organic Chemistry Simplified, by R. Macy (2nd ed.), 611 pp. Chemical Publishing Co., Inc., New York, 1955. Price, $12.00.

The plan of this book is different from that of most standard organic chemistry textbooks in that it aims to explain why various reactions take place, rather than simply cataloguing the classes of compounds with their reactions. The main emphasis is placed on the development of structures of the various types of compounds on the basis of the electron theory.

The book is divided into four parts: the unique position of the carbon atom in chemistry, the architecture of carbon compounds, the classification of carbon compounds, and special topics in organic chemistry. The first two parts emphasize strongly the current concepts of molecular and electronic structure. The last two parts of the book cover the chemistry of the various classes of compounds from the simple alcohols and halogen compounds to such materials as hormones, vitamins, and chemical warfare agents.

While the book would certainly not be classed as an advanced text, it might be a useful addition to the library of those chemists who graduated before the development of the newest electronic concepts in organic chemistry.

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Official Methods of Analysis of the Association of Official Agricultural Chemists, 8th ed., 1008 pp. Association of Official Agricultural Chemists, Inc., P. O. Box 540, Benjamin Franklin Station, Washington 4, D. C., 1955. Price, $12.00 domestic; $12.50 foreign.

Official Methods of Analysis of the A.O.A.C. contains the standardized methods

used by governmental agencies at the federal, state, and municipal levels which are concerned with food and drug regulations, by the industries to which these regulations apply, by governmental and private organizations in establishing and checking specifications for various materials, and by research workers in agriculture.

The methods of analysis are adopted only after collaborative studies demonstrate that they are convenient and give reproducible results in the hands of qualified analytical chemists. In this eighth edition, the Association presents its methods as revised up to March 17, 1955, since the publication of the seventh edition in 1950. The current edition has been expanded by approximately 100 pages, principally in the chapters which deal with pesticides, flavoring extracts, drugs, extraneous materials, microchemical methods, and nutritional adjuncts, including vitamins. A chapter on spectroscopic methods and a section on hormone drugs have been added.

The book contains 42 chapters and a general index. It comprises six major divisions: (1) soils, fertilizers, and related materials; (2) miscellaneous materials which are not foods or drugs; (3) foods; (4) drugs and cosmetics; (5) general methods (microbiological, microchemical, radioactive, and spectroscopic); and (6) reference tables.

Of particular interest to cereal chemists and technologists are the chapters on baking powders and baking chemicals, cereal foods, drug products, eggs and egg products, flavoring products, grain and stock feeds, oils, fats, and waxes, preservatives, spices and other condiments, coloring matters, extraneous materials, and those dealing with microbiological, microchemical, and spectroscopic methods and the preparation of standard solutions. This book is indispensable to chemists in agriculture and in the food and drug industries.

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Cereal Chemistry

EDITORIAL POLICY

Cereal Chemistry publishes scientific papers dealing with raw materials, processes, or products of the cereal industries, or with analytical procedures, technological tests, or fundamental research, related thereto. Papers must be based on original investigations, not previously described elsewhere, which make a definite contribution to existing knowledge.

Cereal Chemistry gives preference to suitable papers presented at the Annual Meeting of the American Association of Cereal Chemists, or submitted directly by members of the Association. When space permits, papers are accepted from other scientists throughout the world.

The papers must be written in English and must be clear, concise, and styled for **Cereal Chemistry.**

Manuscripts for publication should be sent to the Editor in Chief. Advertising rates may be secured from and subscriptions placed with the Managing Editor, University Farm, St. Paul 1, Minnesota. Subscription rates, $11.00 per year. Foreign postage, 50 cents extra. Single copies, $2.50; foreign, $2.60. Back issues, $3.00.

SUGGESTIONS TO AUTHORS

General. Authors will find the last volume of *Cereal Chemistry* a useful guide to acceptable arrangements and styling of papers. "On Writing Scientific Papers for Cereal Chemistry" (*Trans. Am. Assoc. Cereal Chem.* 6:1–22. 1948) amplifies the following notes.

Authors should submit two copies of the manuscript, typed double spaced with wide margins on 8½ by 11 inch white paper, and all original drawings or photographs for figures. If possible, one set of photographs of figures should also be submitted. Originals can then be held to prevent damage, and the photographs can be sent to reviewers.

Editorial Style. A.A.C.C. publications are edited in accordance with *A Manual of Style*, University of Chicago Press, and *Webster's Dictionary*. A few points which authors often treat wrongly are listed below:

Use names, not formulas, for text references to chemical compounds. Use plural verbs with quantities (6.9 g. were). Figures are used before unit abbreviations (3 ml.), and % rather than "per cent" is used following figures. All units are abbreviated and followed by periods, except units of time, which are spelled out. Repeat the degree sign (5°–10°C.). Place 0 before the decimal point for correlation coefficients ($r = 0.95$). Use * to mark statistics that exceed the 5% level and ** for those that exceed the 1% level; footnotes explaining this convention are no longer required. Type fractions on one line if possible, e.g., $A/(B + C)$. Use lower case for farinograph, mixogram, etc., unless used with a proper name, i.e., Brabender Farinograph. When in doubt about a point that occurs frequently, consult the Style Manual or the Dictionary.

For more detailed information on manuscript preparation see
Cereal Chem. **30:** 351–352 (1953).

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International Symposium on Cereal Grains

During the 41st Annual Meeting of the American Association of Cereal Chemists
Hotel Commodore ★ *New York City* ★ *May 20-24, 1956*

★

An international symposium on cereal grain will be held during the 41st Annual Meeting of the American Association of Cereal Chemists in New York City in 1956.

★

It is felt that such a symposium would serve to bring together, possibly for the first time, many scientists engaged in the aspects of supplying a significant, if not the most significant, portion of the world's food to exchange ideas, practices, and knowledge.

★

This is the first national convention of the A.A.C.C. to be held in New York since 1949. It will afford a good geographical opportunity particularly for scientists from Western European countries to attend.

★

Of special interest to visitors will be the fact that the scientists from the experimental stations and research laboratories of the United States Department of Agriculture plan to present a series of papers.

★

In general, the Program Committee would like the symposium to cover the problems of planting, harvesting, and utilizing the cereal crops.

★

Any area of significance in this broad field will be a worthy subject for a paper.

★

Such areas might include the problems of wheat and other grain genetics, soil agronomy, insect control, fertilization, harvesting, storage, milling, baking, and chemical and physical research in connection with any of these problems.

★

Papers presented might include any specialized areas within the broad outline above, or might consist of a review of a particular country's problems in relationship to any portion of the above.

★

The American Association of Cereal Chemists is a professional organization of people engaged in these areas in the United States, Canada, and elsewhere, and as such does not have funds to finance such a program. It is hoped that the institutions, the governments, and organizations sponsoring such research might find it possible to send people to this international symposium.

★

Members desiring to present papers to the convention should communicate with the Program Chairman, Gaston Dalby, Ward Baking Company, New York 54, New York. Local arrangements are under the chairmanship of Enrico Palomba, Anheuser-Busch, Old Bridge, New Jersey.

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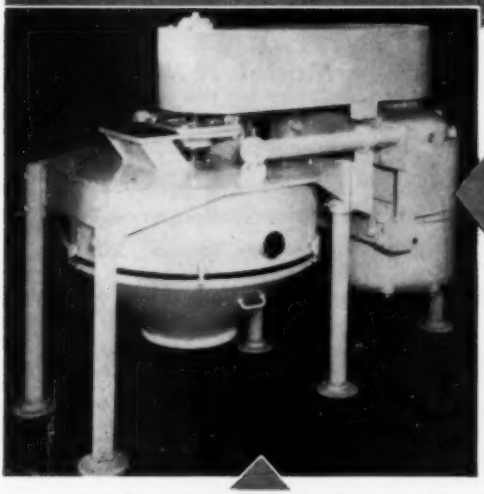

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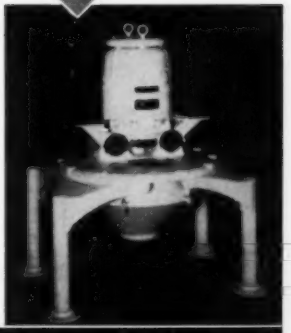

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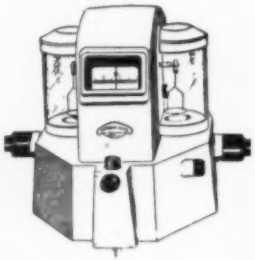

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These levels will be applied in random sampling of cars of wheat.

Excerpt from U.S. Health, Education and Welfare Food and Drug Administration Bulletin M2715.

New Levels Effective During 1956 Crop Year

WASHINGTON, D. C. — The Food and Drug Administration has announced that it will tighten sanitary requirements on wheat beginning July, 1956 to promote increased cleanliness in the Nation's food supply.

George P. Larrick, Commissioner of Food & Drugs, stated that during the period January through July 31, 1955 wheat from 3754 cars was examined. Of these, 29 cars were found to contain filth in excess of the rodent actionable level, and 3 cars showed insect damage to the extent of 2 per cent or more of the kernels.

The Commissioner said: "Under the Program, substantial progress has been made in improving handling and storage of wheat to prevent contamination. However, experience acquired during the past six months shows that the levels of judgment should be tightened if the program is to continue to be effective."

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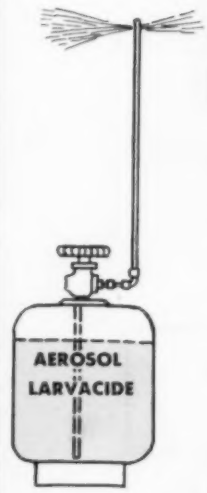

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